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CONTRACT QUALITY ASSURANCE PROJECT PLAN RICHARDS GEBAUR AIR FORCE BASE KANSAS CITY MO

3/31/1995

DAMES & MOORE

CONTRACT QUALITY ASSURANCE PROJECT PLAN RICHARDS-GEBAUR AIR FORCE BASE

F-41624-94-D-8102
DELIVERY ORDER 0001
PREPARED FOR AIR FORCE CENTER FOR ENVIRONMENTAL EXCELLENCE

BROOKS A.F.B. TEXAS

March 31, 1995

PREPARED BY
DAMES & MOORE, INC.

CONTRACT GENERIC QUALITY ASSURANCE PROJECT PLAN

RICHARDS-GEBAUR AIR FORCE BASE
F41624-94-D-8102-0001
REVISION 0
MARCH 9, 1995

PREPARED FOR
AIR FORCE CENTER FOR ENVIRONMENTAL EXCELLENCE
BROOKS A.F.B., TEXAS

Prepared by
DAMES & MOORE, INC.

Approved by:

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Dave Van Dyke — 3/28/95
Dames & Moore Delivery Order Manager, Dave Van Dyke — Date

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ACRONYMS

ABN	Acid/Base Neutral Extractable Compounds
ADC	Air Defense Command
AFB	Air Force Base
AFBCA	Air Force Base Conversion Agency
AFCEE	Air Force Center for Environmental Excellence
AFCS	Air Force Communications Services
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing Materials
ATC	Air Traffic Control
BH	Soil Boring
BRAC	Base Realignment and Closure
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Program
COC	Chain of Custody
CQP	Construction Quality Plan
DO	Delivery Order
DO	Dissolved Oxygen
DQO	Data Quality Objectives
DU	Matrix Duplicate
EIS	Environmental Impact Statement
FAA	Federal Aviation Administration
FB	Field Blank
FD	Field Duplicate
FFA	Federal Facility Agreement
FS	Feasibility Study
FSP	Field Sampling Plan
GC/FID	Gas Chromatograph/Flame Ionization Detector
GC/MS	Graphic Chromatograph/Mass Spectrometry
GFAA	Gas Furnace Atomic Absorption
GSA	General Services Administration
HSP	Health and Safety Plan
ICAP	Inductively Coupled Argon Plasma Atomic Mission Spectrometry
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
ITIR	Informal Technical Information Report

ACRONYMS

IRP	Installation Restoration Program
KCI	Kansas City International Airport
LCS	Laboratory Control Sample
MAC	Military Airlift Command
MB	Method Blank
MDL	Method Detection Limits
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MW	Monitoring Well
NEPA	National Environmental Policy Act
NHPA	National Historic Preservation Act
NPL	National Priorities List
NTU	Nephelometric Turbidity Units
OU	Operating Unit
PA	Preliminary Assessment
PCB	Polychlorinated Biphenyls
PID	Photoionization Detector
PRP	Potentially Responsible Party
PZ	Piezometer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QPP	Quality Program Plan
RA	Remedial Action
RD	Remedial Design
RI	Remedial Investigation
RPD	Relative Percent Difference
RW	River Water
SAP	Sampling and Analysis Plan
SC	Site Characterization
SCS	Single Control Sample
SD	Sediment
SHPO	State Historic Preservation Office

ACRONYMS

SI	Site Inspection
SOP	Standard Operating Procedure
SOW	Statement of Work
TB	Trip Blank
TCLP	Toxicity Characteristic Leaching Procedure
TICS	Tentatively Identified Compounds
TOC	Total Organic Content
TR	Technical Report
TPH	Total Petroleum Hydrocarbons
UPS	United Parcel Service
USC	United States Code
USCS	Unified Soils Classification System
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
TDS	Total Dissolved Solids

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1.0 PROJECT DESCRIPTION

Richards-Gebaur Air Force Base (AFB), Figure 1, is an Air Force Base Conversion Agency (AFBCA) base located in west-central Missouri, approximately 18 miles south of downtown Kansas City and about 3 miles east of the Kansas state line. Base property presently is comprised of about 428 acres. Associated with this acreage is about 421 acres of easements, Figure 2.

Richards-Gebaur AFB was recommended for closure by the 1991 Defense Base Closure and Realignment Commission. The Commission's recommendations were accepted by the President and submitted to Congress on July 12, 1991. As Congress did not disapprove the recommendations in the time given under the Base Realignment and Closure (BRAC) Act of 1990 (Public Law (P.L.) 101-510, Title XXIX), the recommendations have become law. Richards-Gebaur AFB closed on 30 September, 1994.

The Air Force is required to comply with the National Environmental Policy Act (NEPA), 42 U.S. Code (U.S.C.) §54321 et seq., in the implementation of base disposal and reuse. The Air Force must make a series of interrelated decisions concerning the disposition of base property. An environmental impact statement (EIS) was prepared to provide information on the potential environmental impacts resulting from the disposal and proposed reuse of the base property. The Federal Aviation Administration (FAA) was a cooperating agency in the preparation for the EIS. The FAA makes decisions on its own and assists the Air Force in making related decisions concerning Richards-Gebaur AFB property. Several alternative reuse concepts are under study to identify the range of potential direct and indirect environmental consequences of disposal.

The Installation Restoration Program (IRP) was initiated at Richards-Gebaur in 1982 with a preliminary assessment of past waste disposal practices. Since that time, multiple studies have identified seven IRP sites (on AFBCA property). Preliminary assessment work is complete at six of the seven IRP sites. Preliminary assessment work is in progress at one IRP site. SC work is complete at two of the seven IRP sites. SC is in progress at five IRP sites. RE work is in progress at one IRP site. One IRP site is undergoing site closure (RI/FS work was not required). The only IRP site with SC evaluation underway is the Fire Valve Area-SS009.

Additionally SC is planned for four IRP sites. Physical evidence (not just sampling) can also trigger a preliminary assessement/site characterization.

Delivery Order (DO) 0001 of the Statement of Work (SOW) defines the scope of the basic contract Quality Program Plan (QPP) to be developed for the remedial actions to be performed at Richards-Gebaur AFB, at waste disposal and spill sites that pose a threat to human health and the environment. The QPP encompasses a wide range of methods and technologies supporting activities necessary to remedy contaminated site conditions with differing technical and regulatory requirements. Both proven and innovative technologies and methods may be needed to accomplish the remedial actions that this QPP will support. The QPP includes the Environmental Health and Safety Plan (HSP), the Construction Quality Plan (CQP), and the Environmental Sampling and Analysis Plan (SAP). The SAP includes a Field Sampling Plan (FSP) and a Quality Assurance Project Plan (QAPP). Future site specific QPPs will be based upon this approved basic contract QPP.

This Quality Assurance Project Plan (QAPP) presents an example of the organization, objectives, functional activities, and quality assurance (QA) and quality control (QC) activities that may be associated with future site specific evaluations. This QAPP also describes the protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory and field analyses.

This section describes a general field investigation and includes site background information, generic project objectives and scope, a possible sampling network and rationale, data quality objectives (DQOs), and project schedule.

1.1 INTRODUCTION

This QAPP has been prepared on behalf of the Air Force Center for Environmental Excellence (AFCEE) by Dames & Moore and Chemron Incorporated Mound Valley, 10526 Gulfdale, San Antonio, Texas 78216.

The requirement for and scope of this QAPP was delineated in the SOW of Contract No. F41624-94-D-8102 Delivery Order 0001, dated 1 September, 1994. This QAPP includes the format and content required by the SOW and follows USEPA guidance for conducting Remedial Investigations and Feasibility Studies under CERCLA. This QAPP presents a general scope and rationale of possible field and lab measurements potentially planned for future work at Richards-Gebaur AFB. A Field Sampling Plan also has been prepared and submitted and is entirely incorporated into this QAPP through reference.

1.1.1 Overall Project Objectives

The objective of the DO is to protect the public health and the environment to the extent necessary through prevention or reduction of release or migration of hazardous wastes or hazardous constituents to the ground water, surface water, sediment, and soils in and around Richards-Gebaur AFB, during remedial actions to be performed at waste disposal and spill sites. The objective will be achieved through RI and remediation phases. The RIs will evaluate the nature, extent, direction and rate of movement of release of hazardous waste or hazardous constituents at or from the AFB to the ground water, surface water, sediment, and soils. The remediation will identify and evaluate alternatives for the methods and technologies supporting activities necessary to remedy contaminated site conditions.

To comply with the primary purpose of the DO, the site characterization through remediation will be conducted with the following objectives:

1. Verify and evaluate the type of contamination in previously identified waste disposal sites and spill sites.

2. Evaluate the nature and extent of regulated chemical contamination in previously uninvestigated areas (including soil, ground water, surface water and sediment), as these media are potential contaminant transport pathways.

3. Collect sufficient hydrogeological, geotechnical, and water quality data on contaminated site media to develop and evaluate remedial measures.

4. If necessary, collect sufficient chemical and physical data to conduct a baseline risk assessment.

This FSP will document the procedures Dames & Moore will use to conduct site investigation that will assess:

1. The presence or absence of total petroleum hydrocarbons (TPH) and any other hazardous wastes or hazardous constituents;

2. The nature and extent, and rate of movement of TPH and other hazardous wastes or hazardous constituents.

3. The possible routes of migration of TPH and other identified hazardous wastes or hazardous constituents, including a characterization of the geology and hydrogeology of the AFB which delineates possible routes of migration;

4. The extent and potential for migration of TPH and other identified hazardous wastes or hazardous constituents, through each of the environmental media; and

5. Support the development of alternatives from which a remedial measure will be selected by the BRAC Cleanup Team.

1.1.2 Project Status/Phase

Dames & Moore shall develop the basic contract QPP to support the implementation of remedial actions as specified in the basic contract and in accordance with compliance documents listed in the SOW paragraph 2.1. In this approach, the data collected during this SC assessment phase will influence the development of remedial alternatives, which may, in turn, affect the data needs and scope of subsequent phases of field investigations.

This first phase of the investigation may include:

- Subsurface soil sampling,
- Ground water sampling,
- Surface water sediment sampling,
- Laboratory analyses of TPH and other parameters,
- Field screening for total volatile organic compounds (VOCs),
- Geotechnical sampling,
- Installation of wells and/or piezometers,
- Tank area soil sampling, and
- Background/upgradient sampling in all media.

Data from the initial phase will be evaluated qualitatively and quantitatively to assess if data gaps exist or if data quality issues affected data completeness. The SC report will be prepared and submitted to AFCEE. The rationale, scope, and data quality objectives of any subsequent investigations will then be presented to and discussed with AFCEE.

When sufficient data of acceptable quality have been collected to perform site characterization, the baseline risk assessment (if necessary) and remedial alternatives study will be conducted by Dames & Moore and presented to AFCEE.

1.1.3 QAPP Preparation Guidelines

All QA/QC procedures described herein are structured in accordance with applicable technical standards, and USEPA requirements, regulations, guidance, and technical standards. This QAPP was prepared in accordance with the AFCEE Handbook for the Installation Reservation Program (IRP) Remedial Investigation and Feasibility Studies (RI/FS), September, 1993, and a USEPA guidance manual, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, (QAMS)-005-80.

1.2 SITE DESCRIPTION

Richards-Gebaur AFB is an Air Force Base Conversion Agency (AFBCA) base located in west-central Missouri, Jackson and Cass Counties, and about 3 miles east of the Kansas state line. A brief description of the facility, its location, geological setting, and associated features is presented below.

1.2.1 Location

Richards-Gebaur AFB is located approximately 18 miles south of downtown Kansas City, Missouri. The northern portion of the base is located in Jackson County and the southern portion of the base is located in Cass County. Primary access to the base is by U.S. Highway 71.

1.2.2 Base Size

The base property is currently comprised of about 428 acres in 11 parcels. Associated with this acreage is about 421 acres of easements. The Cantonment Area, covering 209 acres, is the largest parcel and contains the main aviation support and administration areas. Nine smaller parcels, ranging from 1 to 13 acres, surround the Cantonment Area. The Belton Training Complex, about 4 miles south of the Cantonment Area, encompasses 184 acres and is largely undeveloped.

1.2.3 Natural and Manmade Features

In compliance with the National Historic Preservation Act (NHPA), the Air Force initiated a review process with the Missouri State Historic Preservation Officer (SHPO). A records and literature search were performed in April,1993, using environmental and cultural resources documents from the SHPO's office and Richards-Gebaur AFB.

Surveys conducted in 1977 and 1979 concluded that there were no prehistoric archeological sites of significance identified on Richards-Gebaur AFB. A review of real property records in April 1993 indicated that the remaining built environment within the area of potential effect at Richards-Gebaur consists of approximately 83 buildings and structures; of

these, none have yet attained the age of 50 years. Historical research, including interviews with the base historian and other individuals familiar with the history of the installation, preliminarily indicated that only one building, Building 602, is associated with events or persons significant in the past and the Missouri SHPO has determined that this building is potentially eligible to the National Register.

Traditional resources, such as archeological sites, burial sites, ceremonial areas, caves, mountains, water sources, plant habitat or any other natural area important to a culture for religious or heritage reasons, are potentially protected by the same regulations and afforded the same protection as other types of historic properties. Modern traditional resources at the base would most likely be associated with various Indian tribes; however, no such resources have been identified. There are no known important fossil localities at the base and no lands are set aside for fossil preservation.

1.2.4 Topography

Richards-Gebaur AFB is located within the Osage Plains region of the Central Lowland physiographic province. The region is characterized by low relief, wide, maturely dissected uplands, and relatively steep valley slopes.

Ground contours will be shown on the figures accompanying the FSP for specific DOs. The topography of Richards-Gebaur AFB is gently rolling with an elevation range between 1,060 and 960 feet above mean sea level. Most of the base storm water drains into the Little Blue River with the exception of the Belton Training Complex, which drains into the West Fork of East Creek. Both of these watersheds ultimately flow into the Missouri River.

1.2.5 Local Geology and Hydrogeology

The geology of the base is characterized by thin loess deposits over residual soils derived from the in-situ weathering of the underlying limestones and shales. The soils belong to the Macksburg-Urban series, which is defined as being poorly drained silt and silt clay loams, covered in places by urban features. Rock outcrops are found along Scope Creek include the Argentine Limestone Member of the Wyandotte Formation, the Lane Formation, the Raytown Limestone Member of the Iola Formation limestone, and the Chanute Formation. The Argentine

Member is a light gray limestone characterized by thin, wavy bedding, except in the lower few feet, where the unit is thick-bedded. The Lane Formation is a medium gray to bluish gray shale that is commonly silty in the upper part. The Raytown Member is a medium bluish gray, wavy bedded limestone, locally containing interbedded lenses of shale approximately 3 inches thick. The Chanute Formation is a gray, red, purplish red, and green shale with thin nodular limestone near the middle, and local occurrences of cross bedded sandstone and conglomerate. All of the exposed units are Pennsylvanian in age. The weathered zone overlying these rocks (in the undisturbed state) is typically 2 to 15 feet thick. The soil is generally fine silty clay with a hydraulic conductivity of approximately 10^{-7} centimeters per second. The depth to ground water is generally shallow, but varies seasonally, with topography, and the variance is highly dependent on the number and composition of the perched aquitards in the local area.

1.3 SITE/FACILITY HISTORY

1.3.1 General History

In 1941, portions of the land now occupied by Richards-Gebaur AFB were acquired by Kansas City for use as an auxiliary airport (Grandview Airport). In 1952, the Aerospace Defense Command leased the airport from the city for air defense operations, and in 1953 the property (approximately 2,400 acres was formally conveyed to the United States government for establishment of an Air Force base. The C-46 airlift aircraft were the original Air Force aircraft stationed at the base. Conversion to C-119 and C-124 aircraft occurred in 1957 and 1961, respectively. In 1957, the base was named Richards-Gebaur AFB.

Until 1970, the Air Defense Command (ADC) had the primary mission on base. In 1970, the Air Force Communications Service (AFCS) relocated its headquarters from Scott AFB, Illinois, to Richards-Gebaur AFB and assumed command. In 1971, the C-124 reciprocating engine aircraft were phased out and replaced with C-130 aircraft. It is reported that this conversion reduced the industrial waste produced by the base as well as reducing the generation of waste oil in half. AFCS moved back to Scott AFB in 1977 and Richards-Gebaur AFB came under the Military Airlift Command (MAC).

AFRES assumed operation control of the base in October 1980. In 1981, around 80 percent of the base property (including runways and taxiways) was excessed (transferred) to the

General Services Administration (GSA). The GSA then transferred a majority of the air port-related property to Kansas City Aviation Department as a public benefit transfer with the condition of continued runway access (for a fee) by the Air Force. Other excessed parcels were also transferred by GSA for public and other military uses to Kansas City, Federal Aviation Administration, city of Belton, the Department of the Navy, and the Department of the Army. Base property presently is comprised of about 428 acres. Associated with this acreage is about 421 acres of easements. Richards-Gebaur AFB closed on 30 September 1994.

1.3.2 Past Data Collection Activities

The Installation Restoration Program (IRP) was initiated at Richards-Gebaur in 1982 with a preliminary assessment of past waste disposal practices. Since that time, multiple studies have identified seven IRP sites (on AFBCA property). Preliminary assessment work is complete at six of the seven IRP sites. SC work is complete at two of the seven IRP sites. SC is in progress at five IRP sites. RE work is in progress at one IRP site. One IRP site is undergoing site closure (RI/FS work was not required). The only IRP site with SC evaluation underway is the Fire Value Area-SS009. Additional SC is planned for four IRP sites. Physical evidence (not just sampling) can also trigger a preliminary assessment/site characterization.

1.3.3 Current Status

No permitted RCRA facilities are present or required by law at Richards-Gebaur AFB. Richards-Gebaur AFB has no sites on the NPL and subsequently has not entered into a FFA.

Compliance activities at Richards-Gebaur AFB are being coordinated with environmental restoration activities under the IRP when necessary. The base does not require or have any RCRA-permitted facilities for waste storage, and does not treat hazardous waste on site. The base generates enough hazardous waste to be classified as a small quantity generator only one or two months out of a year. For the remainder of the year, the base is classified as a conditionally exempt small quantity generator. Compliance activities address the management of petroleum products, hazardous materials, hazardous waste, asbestos, solid waste, pollution prevention, water quality, air quality, pesticides, polychlorinated biphenyls (PCBs). etc. Corrective actions and closure activities will remain compliance issues regardless of size or scope.

1.4 PROJECT OBJECTIVES

The project objectives of this SC are to collect data of sufficient quality to support the DO. These requirements are to delineate site contamination by petroleum hydrocarbons and other compounds; to assess if releases from the site, or a substantial threat of release of hazardous substances from the site, present a risk to human health and the environment; and to use these data in the development and evaluation of remedial measures.

1.4.1 Specific Objectives and Associated Tasks

The purpose of specific DO SCs is to gather sufficient information on the nature and extent of the contamination to develop and evaluate viable remedial measures for specific sites and perform a baseline risk assessment, if necessary.

The specific objectives of SCs data collection activities may include some of the following tasks:

- Evaluate the nature and extent (horizontal and vertical) of petroleum hydrocarbon contamination in site soil and ground water during well and piezometer installation, and ground water sample collection.

- Measure physical and chemical parameters (such as Total Organic Carbon, (TOC) Total Dissolved Solids, (TDS) pH,) that may affect potential ground water monitoring, remediation, and barrier/containment measures.

- Assess existing surface water quality upstream, downstream, and on site through analysis of grab samples and sediment pore water.

- Establish upgradient and background conditions in soil, sediment, surface water, and ground water for the site.

- Qualitatively investigate the nature and extent of contamination from spills in soils from previously documented spill areas.

- Evaluate if leakage from on-site storage tanks has occurred.

- Verify and further define the nature and extent of contamination of previously identified on-site areas, surface water, and sediments.

- Evaluate the physical characteristics of the hydrogeologic units beneath the site to assess potential contaminant migration, and physical and chemical parameters that may affect potential ground water monitoring, remediation, barrier or containment technologies.

1.4.2 Project Target Parameters and Intended Data Usages

The project target parameters will be chosen based on a knowledge of the wastes released at the site (including documentation regarding on-site spills), contaminants identified during previous environmental investigations at the site, and those necessary to comply with the state and USEPA regulations. Table 1 (format only) presents a general outline/list of possible laboratory analytical parameters, field parameters, the location and depth of samples, and the rationale for the selection of sampling location and intended analyses.

The data collected during the SC will be used to further evaluate the magnitude, extent, and type of contamination at the site. The data will be compared to background soil, sediment, and water concentrations detected in background samples obtained upgradient of known areas of site contamination. Additionally, the data collected during the SC will be used to predict contaminant migration in the soils, sediments, surface water, and ground water. This information then will be used to perform a baseline risk assessment, if necessary, and will be used to perform a preliminary screening of potential remedial technologies that are applicable based on the site conditions.

Areas selected to obtain background samples will be selected because they are similar to the study area in terms of physical makeup, soil type, and stratigraphy. Planned background locations will be chosen based on distance from the source and similarities in pedology, stratigraphic layers, and historic information, and are described in Section 1.5.3.

The planned number of background locations by media are listed in Table 2 (format only) and will be derived from guidance contained in *Soil Sampling Quality Assurance User's Guide* (USEPA 600/8-89/046, March 1989). Definitive guidance on the exact number of statistically appropriate background locations is based on a knowledge of the cleanup/action level to be used. As these action/cleanup levels have not yet been determined for this SC, the following assumptions have been made:

1. A Type II error (false negative) is considered to be of greater importance at the site than a Type I error.

2. The confidence level ($1-\alpha$) appropriate for a site investigation is 80 percent.

3. The power ($1-\beta$) is 95 percent.

4. The relative increase over background that is reliably detectable based on field and lab imprecision with a probability of 95 percent is 30 percent.

5. The number of background samples is determined from a one-sided, one-sample t test to achieve a minimum detectable 30 percent relative difference at an 80 percent confidence level and a power of 95 percent.

6. The expected coefficient of variation of TPH concentrations in the sampling media was approximated as follows: soil 20 percent, sediment 20 percent, surface water 15 percent, ground water 10 percent.

1.4.2.1 Field Parameters

The following parameters will be measured on site during the field investigation:

- Unified Soils Classification System (USCS) soil and sediment classification, including color (Munsell), consistency, structure, mottling, layering, lenses, fractures, organic matter or voids.

- Photoionization Detector (PID) screening of total VOCs.

- Ground water temperature, pH, turbidity, and conductivity.

1.4.2.2 Laboratory Parameters

Specific laboratory parameters and their reporting limits will be presented in Table 3 (format only). The corresponding level of field and laboratory quality control is presented in Table 4, Sampling and Analysis Summary Table for the Richards-Gebaur SC.

1.4.3 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during the SC activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels which address various data uses and the methods required to achieve the desired level of quality. The level selected for each media and analyte in this SC are listed in Table 4 (format only). The levels cited are:

- Screening (DQO Level 1): This provides the lowest data quality, but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). For this site, data generated under DQO Level 1 will be for health and safety monitoring and include total organic vapor monitoring by PID. Health and safety monitoring activities applicable to these measurements are described in the site-specific health and safety plan (HSP) and are not covered in this QAPP.

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile laboratory generated data depending on the level of QC. Data generated in the field under DQO Level 2 will consist of pH, turbidity, conductivity, and temperature in surface water and ground water.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Under the direction of the Contracting Officer, Dames & Moore has overall responsibility for conducting all phases of the Remedial Action (RA). Dames & Moore will provide project management, perform the field investigations, prepare the SC report, and direct the subsequent remedial measures. The review of all environmental and hydrogeologic data will be conducted by the Dames & Moore Site Quality Assurance Manager, DO Manager, Field Team Leader and Project Geologist. The various quality assurance, field, laboratory and management responsibilities of key project personnel presented in Figure 5 (possible format) are defined below.

2.1 PROJECT ORGANIZATION CHART

The lines of authority for the Richards-Gebaur SC project can be found in Figure 5. The responsibilities of all personnel shown on this figure are defined below.

2.2 MANAGEMENT RESPONSIBILITIES

2.2.1 Contracting Officer

The Contracting Officer is sole authority to authorize change price, quantity, quality, place of performance, schedule, or any other terms or conditions of the contract or delivery orders.

2.2.2 USEPA Region VII

The State of Missouri and EPA Region VII provide the standard and regulatory basis for remediation at the sites.

2.2.3 AFCEE/ERB Project Manager

The AFCEE/ERB Project Manager as the Contracting Officer's Representative (COR) is responsible for the technical monitoring of contract performance and is the technical point of contact for this effort. As the appointed COR, the AFCEE/ERB Project Manager will:

- Be responsible for the technical monitoring of Contractor performance, and will act as the technical point of contact for this effort.
- Expedite the technical reviews of the Contractor's proposal for any changes to the DO.
- Authorize release of materials of actions requiring Government concurrence, as specified in the DO.
- Coordinate activities with Contractor personnel and other individuals involved in this effort at Richards-Gebaur AFB.
- Expedite the review of invoices/payment vouchers.
- Be responsible for the inspection and acceptance of the completed effort specified in subject DO.
- Maintain written records, for PCO review, of all actions taken by technical personnel and the contractor to ensure that costs, schedule and technical performance is documented.
- Attend meetings, i.e., site visits, pre-performance conferences, as the official Government technical representative, as needed.
- Expedite the evaluation of technical reports submitted by the contractor.

2.2.4 Dames & Moore Program Manager

The Program Manager is Mr. Gary Alkire, Senior Engineer with the Dames & Moore San Antonio, Texas office. Mr. Alkire is responsible for project oversight from both a technical

and managerial perspective, and is ultimately responsible for meeting project objectives in terms of scheduling, technical accomplishments, and budgetary restraints.

2.2.5 Dames & Moore Delivery Order Manager

The Dames & Moore DO Manager (to be determined) has responsibility for day-to-day activities to ensure the project, as defined in the project documents (FSP, QAPP and HASP), meets AFCEE/ERB objectives and Dames & Moore quality standards. The Dames & Moore DO Manager will provide assistance to the AFCEE/ERB Project Manager in terms of writing and distributing the QAPP to appropriate parties connected with the project (including the Dames & Moore physical testing laboratory, Chemron Inc., R & R International, Inc., and EnviroKlean). The Dames & Moore DO Manager will report directly to the Dames & Moore Program Manager and will coordinate with the AFCEE/ERB Project Manager and the Air Force Base Conversion Agency. The DO Manager is responsible for technical quality and project oversight.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

2.3.1 AFCEE/ERB Contracting Officer's Representative

The AFCEE/ERB COR is responsible for auditing the implementation of the QA program in conformance with the project QPP and USEPA requirements. Specific functions and duties include:

- Providing QA audits on various phases of the field operations;
- Reviewing and approving QA plans and procedures;
- Providing QA technical assistance to Dames & Moore project staff and subcontractors;

2.3.2 Dames & Moore QA Manager

The Dames & Moore QA Manager is responsible for ongoing surveillance of project activities to help ensure conformance to this QAPP and to evaluate the effectiveness of it's requirements. The QA Manager has access to all personnel and subcontractors as necessary to resolve quality problems. The QA Manager has the authority to stop lab or field activities if major deficiencies in quality occur. The QA Manager will be the primary technical point of contact with the laboratory QA officers on data quality issues. The QA Manager will be responsible for:

- Approving and implementing this QAPP;
- Providing for retention of QA records;
- Participating in the QA audits of lab and field activities;
- Providing QA reports to the Dames & Moore DO Manager on the result of audits and the need/status of corrective actions;
- Developing and initiating corrective and preventive actions in coordination with the DO Manager;
- Recommending changes, as appropriate, to improve the effectiveness of this QAPP;
- Reviewing data assessment reports received from Dames & Moore validation personnel against the project objectives and DQOs;
- Providing input to the monthly QA reports, and providing interim verbal QA reports to the Dames & Moore Program Manager and Corporate Executive; and
- Preparing the QA section of the draft and final SC reports.

The Dames & Moore QA Manager reports directly to the Dames & Moore Corporate Executive and will be responsible for ensuring that all Dames & Moore procedures for this project are being followed. In addition, the Dames & Moore QA Manager will be responsible for the data validation of all sample results from the analytical laboratory.

2.3.3 Site Health and Safety Officer

The Site Health and Safety Officer will be responsible for assuring that all team members adhere to the health and safety requirements described in the H&S Plan. Additional responsibilities of the Health and Safety Officer are as follows:

1. Updating equipment or procedures based upon new information gathered during the site inspection.

2. Modifying the levels of protection based upon site conditions.

3. Determining and posting locations and routes to medical facilities and arranging for emergency transportation to medical facilities.

4. Notifying local public emergency officers, including police and fire departments, of the nature of the team's operations and posting their telephone numbers.

5. Examining work-party members for symptoms of exposure or stress.

6. Providing emergency medical care and first aid as necessary. He also has the responsibility to stop any field operation that threatens the health or safety of the team or the surrounding populace.

The Site Health and Safety Officer reports directly to the Dames & Moore Corporate Executive and will be responsible for ensuring that all Dames & Moore procedures for this project are being followed.

2.4 LABORATORY RESPONSIBILITIES

Table 5 (format only) presents the analytical responsibilities by media for each SC laboratory listed below. In general, Chemron (see Figure 6) will be responsible for all laboratory analyses with the exception geotechnical/physical analyses (which will be performed by Dames & Moore).

Dames & Moore Soils Laboratory
150 S. 600 East, Building 3
Salt Lake City, UT 84102-1959
Contact: Eric Rosik (801) 521-9255

Chemron Incorporated
10526 Gulfdale
San Antonio, Texas 78216
Contact: Ronald Oldham (201) 340-8121

Each laboratory will designate a Project or Program Manager, Operations Manager, QA Officer, and Sample Custodian for the project.

2.4.1 Laboratory Project/Program Manager

The laboratory Project/Program Manager will report directly to the Dames & Moore DO Manager and will be responsible for the following:

- Ensuring all resources of the laboratory are available when required; and
- Providing an overview of final analytical reports.

2.4.2 Laboratory Operations Manager

The laboratory Operations Manager will report to the laboratory Project Manager and will be responsible for:

- Coordinating laboratory analyses;
- Scheduling sample analyses;
- Overseeing data review;
- Overseeing preparation of analytical reports; and
- Approving final analytical reports prior to submission to Dames & Moore.

2.4.3 Laboratory Quality Assurance Officer

Chemron's QA Officer has the overall responsibility for data after it leaves the bench. The laboratory's QA Officer will be independent of the operating departments, and will communicate data issues through the laboratory's Project Manager and Dames & Moore's Site QA Manager. In addition, the laboratory's QA Officer will:

- Overview laboratory quality assurance;
- Overview QA/QC documentation;
- Conduct detailed data review;
- Determine whether to implement laboratory corrective actions, if required;
- Notify Dames & Moore QA Manager and Delivery Order Manager of data quality issues;
- Define appropriate laboratory QA procedures; and
- Prepare laboratory standard operating procedures.

2.4.4 Laboratory Sample Custodian

The laboratory's Sample Custodian will report to the laboratory's Operations Manager. Responsibilities of the Sample Custodian will include:

- Receiving and inspecting the incoming sample containers;
- Recording the condition of the incoming sample containers;
- Signing appropriate documents;
- Verifying chain-of-custody and its accuracy;
- Notifying appropriate analytical staff of sample receipt and sample integrity issues (e.g., holding times);
- Assigning a unique identification number and customer number, and entering each into the sample receiving log;
- With the help of laboratory staff, initiating and documenting transfer of the samples to appropriate lab sections; and
- Controlling and monitoring access/storage of samples and extracts.

Final responsibility for project quality rests with the Dames & Moore Program Manager. Independent quality assurance will be provided by the laboratory's Project/Program Managers and QA Officers and the Corporate Executive. The QA Officer will check the quality of their laboratory's work against their SOP's (Appendix A) and the requirements of this QAPP.

2.4.5 Laboratory Technical Staff

The laboratory's technical staff will be responsible for disposition of samples in laboratory, sample analysis, analytical documentation, and identification of corrective actions, if necessary. Laboratory staff will report directly to the laboratory Operations Manager.

2.5 DATA VALIDATION

Dames & Moore will conduct validation studies in accordance with their established procedures as described in Section 9.1 of this QAPP. Data validation will be performed for all analyses which have a Data Quality Objective (DQO) of 3, as shown in Table 4. The QA Project Manager has the responsibility for review of the validation report against this project QAPP and corporate quality standards.

2.6 FIELD RESPONSIBILITIES

2.6.1 Dames & Moore Field Team Leader

The Dames & Moore Field Team Leader is responsible for leading and coordinating the day-to-day field data collection and sampling activities of the various resource specialists under his/her supervision. The Dames & Moore Field Team Leader is a highly experienced environmental professional and will report directly to the Dames & Moore Delivery Order Manager. Specific Field Team Leader responsibilities include:

- Coordinating and integrating site activities.
- Implementing field-related work plans (FSP, H&S Plan), schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing field staff during drilling, well installation and sampling, and supervising the performance of field analyses;
- Implementing QC measures for technical data provided by the field staff, including field measurement data;
- Adhering to work schedules provided by the Dames & Moore DO Manager;
- Writing and approving text and graphics required for field team efforts;

- Coordinating and overseeing technical efforts of drilling subcontractors and surveyors assisting the field team;

- Identifying problems at the field team level, resolving difficulties in consultation with the Dames & Moore DO Managers, implementing and documenting corrective action procedures, and providing communication between field sampling team, Project Managers, and laboratories;

- Participating in preparation of the draft and final SC reports; and

- Providing input to the DO Manager related to schedule and quality issues for monthly QA reports to AFCEE/ERB.

2.6.2 Dames & Moore Field Technical Staff/Field Organization

The Dames & Moore field investigation team will be organized according to the sampling activity to be undertaken. For on-site sampling work, the actual sampling team make-up will depend on the type and extent of sampling and will consist of a combination of the following:

1. Field Team Leader: To be determined for each DO (Dames & Moore)
2. Project Geologist: To be determined for each DO (Dames & Moore).
3. Site Health and Safety Officer: To be determined for each DO (Dames & Moore).
4. Sampling Coordinator: To be determined for each DO (Dames & Moore).
5. Field Sampling Technicians: To be determined for each DO (Dames & Moore).

The Field Team Leader will be responsible for the coordination of all personnel at the site and for providing technical assistance when required. The Field Team Leader or designee will be present whenever sampling occurs, and will keep a general site log which will describe activities conducted on site, include a log of all communications, identify personnel entering and leaving the site, and note general observations regarding site activities (as required by the construction Quality Plan.)

The Project Geologist will be responsible for providing technical supervision of the drilling subcontractor during the installation and development of the monitoring wells. In addition, the Project Geologist will be responsible for the geologic logging and field measurements to be performed during installation of the monitoring wells, drilling of soil borings, and the acquisition of sediment samples.

The Sampling Coordinator will be responsible for the coordination of all sampling efforts and will assure the availability and maintenance of the necessary shipping/packing materials, sample containers, and sampling equipment. The Sampling Coordinator also will: (1) supervise the completion of all sampling documentation; (2) ensure the proper handling and shipping of the samples; (3) be responsible for the accurate completion of the field notebook; (4) provide close coordination with the Dames & Moore Field Team Leader so that laboratories are kept notified of sample shipments; and (5) complete all chain-of-custody forms.

The technical staff (team members) for this project will be drawn from Dames & Moore's pool of corporate resources. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the technical team members will be experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work. All field personnel will adhere to chain-of-custody and field documentation protocols, and will perform field measurements in accordance with Dames & Moore SOPs.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, calibration and preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP.

Analytes of Interest - as defined in the AFCEE Handbook, analytes that are potentially present at the site under investigation or are of concern to the regulators.

Laboratory Control Samples(LCS) - are method blanks spiked with known concentrations of all analytes of interest.

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 Field Precision Objectives

The statistic used to monitor field precision will be RPD as defined by the following equation:

$$RPD = \frac{(Amount\ in\ Field\ Sample - Amount\ in\ Duplicate)}{0.5\ (Amount\ in\ Field\ Sample + Amount\ in\ Duplicate)} \times 100$$

Field sampling precision will be assessed through comparisons of field duplicate sample analytical results. Field duplicate samples (replicates or splits for soil or sediment samples) will be collected at a frequency of 10 percent; one field duplicate will be collected at the location

where the tenth sample is collected. If less than 10 samples are collected for a matrix, a field duplicate will still be collected.

Precision for field measurements will be determined by performing replicate measurements on the same sample, i.e., a second measurement will be done on every tenth sample collected for field measurement.

The frequency for field duplicate collection by matrix and analyte is presented in Table 4. The specific precision objectives for field duplicates and field measurements - by analyte - are presented in Table 6. The source of the limits shown are historical experience and USEPA's definition of "significant differences" for organic data "as a factor of 5 times or greater in the concentration." This factor equates to 133 percent RPD.

Table 6 defines the precision objectives for field duplicates and field measurements for this project.

3.1.3 Laboratory Precision Objectives

Laboratory precision will be assessed by means of replicate analysis. The precision of the method is defined by the control limits established for accuracy (mean value plus or minus three standard deviations of percent recovery). In the analyses of samples in a preparation batch, if the recoveries of analytes in the LCS are within the control limits, the precision is also within limits. Precision can be expressed in percent relative standard deviation (% RSD).

The specific objectives for laboratory precision by analyte and matrix are presented in Table 10.

The sources of the limits are laboratory historical precision data. These precision objectives are sufficient to meet the intended data uses of baseline risk assessment, site characterization, nature and extent of contamination and development of remedial action alternatives.

3.2 ACCURACY

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value. It is presented as the percent recovery of surrogate compounds and/or percent recovery of a known laboratory control sample (LCS) or matrix spike (MS)/matrix spike duplicate (MSD) pair.

3.2.2 Field Accuracy Objectives

Field accuracy is defined as the collection of a sample that truly reflects the quality of the media sampled, is adequately preserved, and is not biased by field induced contamination during sample collection, handling and shipment. Therefore, field accuracy will be assessed by analyzing field blanks and trip blanks, and laboratory checks of pH for preserved samples.

Field blanks will be collected for soil and sediment samples. The collection of the final rinse water from the decontamination of soil or sediment sampling equipment generally is not a good indication of the effectiveness of decontamination procedures when collecting soil or sediment samples.

Since dedicated teflon bailers will be used to sample new and existing wells, there is no possibility of cross contamination between wells, and no field blanks will be collected during well sampling. However, because pumps will be used to develop the wells prior to sampling, field blanks will be prepared at a frequency of one for each day of sampling and will consist of the final rinse water (Type II Reagent Grade) from the decontamination of the pumps. The field blanks will be analyzed for all contaminants of concern at the location of the sampled well.

Trip blanks will accompany every shipment or cooler (whichever is more frequent) which contain samples for VOC analysis. Trip blanks will be prepared by the laboratory, and will accompany all soil, sediment, ground water, and surface water VOC samples during shipment.

The objective is for field and trip blanks to contain no detectable project analytes above the analytical reporting limits, and for all samples to be the proper pH and temperature when received at the laboratory, as indicated in Table 8. However, it is recognized that common contaminants arising from field sampling equipment, decontamination solutions and laboratory operations may be detected including: zinc, iron, sodium, calcium, acetone, methylene chloride, toluene, 2-butanone, and phthalates. The objective for these contaminants will be set at three times the reporting limit; concentrations above these limits detected in field blanks or trip blanks will require the initiation of corrective action for field procedures.

The accuracy objectives for field measurements are stated in Table 9.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of laboratory control samples (LCS) or standard reference materials (SRM) in the form of surrogates and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12.2.1 of this QAPP. The accuracy requirements for the SC analytical services for organics and inorganics reflect Chemron's historical control limits where available these limits are summarized in Table 10 (organics) and 11 (metals). The QA objectives for the remainder of the chemical analyses are presented in Table 12. Accuracy objectives for surrogate recoveries are presented in Table 13.

3.3 COMPLETENESS

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all analytical results and data specified for the project. In addition, all data are compared to project requirements to ensure that specifications were met. Any deviations are reported in the report narrative.

The percent completeness for each set of samples can be calculated as follows:

$$Completeness = \frac{valid\ data\ obtained}{total\ data\ planned} \times 100\%$$

where valid data are determined by the data acceptance criteria defined in this QAPP.

3.3.2 Field Completeness Objectives

Field completeness will be measured as the amount of field measurement (pH, conductivity, temperature, etc.) that comply with field standard operating procedures for calibration and precision objectives compared to all of the field measurements attempted. The field data completeness objective for this project will equal or exceed 90 percent.

Field sample collection completeness will be measured as the total number of field samples of sufficient volume collected for laboratory analysis compared to the total number of samples shown in Table 4. The field sample collection completeness for this project will equal or exceed 95 percent.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness will be assessed as the amount of valid analytical data obtained compared to all analytical data generated. Valid analytical data will be that data which includes, after the data validation process, codes of J, V, X, and A, as shown in Section 9.2.1. Data coded B will only be used if the sample value is greater than 10 times the highest associated field/trip/lab blank. Tentatively Identified Compound data will not be included in percent completeness calculations. Laboratory completeness will be calculated in accordance with the equation shown in Section 12.3. Laboratory completeness for this project will equal or exceed 90 percent, and will be separated by matrix and analyte or analyte group (8260, 8270, 8080, 8150, 6010).

3.4 REPRESENTATIVENESS

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed. Although Chemron strives to accommodate all sample matrices, some samples may require analysis by multiple techniques or using additional cleanup to obtain representative results.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program, and will be satisfied by ensuring that the FSP is followed and proper sampling techniques are used.

During development of this network, consideration was given to existing analytical data, prior investigations, previous remedial activities and constraints inherent to the geologic/hydrogeologic setting of the base. The rationale of the sampling network is discussed in detail in the FSP. Representativeness will be satisfied by ensuring that the FSP is followed, proper sampling techniques are used, proper analytical procedures are followed, holding times of the samples are not exceeded, and sample preservation is adequate.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing field duplicate samples. The sampling network was designed to provide data representative of base conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the Base Closure/Disposal and Reuse program. The rationale of the sampling network is discussed in detail in the FSP.

3.5 COMPARABILITY

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability of data obtained during this phase and subsequent phases of the SC is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used. These new chemical analytical data, however, may not be directly comparable to existing data because of possible differences in procedures and QA objectives in effect at the time the historical data were obtained.

The extent to which the planned geotechnical data is comparable to existing geotechnical data will be determined during data assessment by Dames & Moore.

The procedures used to obtain the planned analytical data, as documented in the QAPP, are expected to provide comparable data. The data will be reported in units consistent with the AFCEE handbook for the IRP guidelines. Comparability between USEPA and Richards-Gebaur SC data bases will also be achieved by using the AFCEE - approved sampling and analysis methods and data formats.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

3.6 SENSITIVITY

The sensitivities for the analytical methods used will attempt to meet the Practical Quantitation limits for the target compound(s) listed in Table 3. It is anticipated that these reporting limits will be achieved for the majority of samples. Higher detection limits may be obtained in oily samples, samples with matrix interferences, and samples containing high concentrations of target and non-target analytes such as Tentatively Identified Compounds (TICS) and soil/sediment samples with high moisture content.

For field analyses, such as pH, conductivity, and temperature, the "detection limit" or sensitivity is specified in the SOPs attached to the FSP.

3.7 LEVEL OF QUALITY CONTROL EFFORT

Field equipment blank, trip blank, duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. As stated in Section 3.2.2, field and trip blanks will be submitted to the analytical laboratory to assess the quality of the data resulting from the field sampling program. Field equipment blank samples will be analyzed to check for procedural contamination during sample collection and handling at the base which may cause sample contamination. Trip blanks will be used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate (and replicate) samples will be analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be collected for every 20 investigative samples. MS/MSD samples are designated/collected for organic analyses only.

The general level of the QC effort will be one field duplicate for every 10 investigative samples. One volatile organic analysis (VOA) trip blank consisting of Type II Reagent Grade water will be included with each shipment of aqueous, soil/sediment VOA samples. Dedicated bailers will be used at all new and existing wells; however, because well development will require the use of the same pump at each well, one field equipment blank will be collected from the final decontamination water of the pump at a rate of one per day of sampling.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and triple the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., ground water, soil). The number of duplicate and field blank samples to be collected are listed in Table 4.

4.0 SAMPLING PROCEDURES

A complete FSP describing the field collection procedures is submitted under separate cover. This QAPP and the FSP form the two portions of the Environmental SAP.

Sample containers and preservatives will be obtained from commercial vendors such as Environmental Sampling Supply, IChem, or Eagle-Picher. All sample containers will be certified as clean by the vendor, and a certificate of analysis will be supplied with each lot ordered. These certificates will be kept on file by Dames & Moore, and a record of the bottle lot numbers and preservatives used in the field will be made in the field sampling log; bottle lots and preservatives will be traceable to the field sample numbers. Sample containers to be used are listed in Table 8, and are grouped by analytes and matrix.

In general, sample collection order will be from the historically least contaminated to the most contaminated areas (upgradient first), and downstream to upstream (surface water first, then sediments), to avoid disturbing the water column. The specifics of the sampling order, by matrix, are referenced in the FSP.

The order of chemical sample collection will be volatiles first, volatile aromaties, then semivolatiles, pesticides/PCBs, and herbicides (if required). Samples for inorganic parameters will be collected next. Geotechnical samples will be collected last.

5.0 CUSTODY PROCEDURES

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is defined as under custody if:

- The item is in actual possession of a person;
- The item is in the view of the person after being in actual possession of the person;
- The item was in actual physical possession but is locked up to prevent tampering; or
- The item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

The sample handling, packaging and shipment procedures summarized below will ensure the samples arrive at the laboratory with the chain of custody intact.

5.1.1 Custody Initiation

1. The Field Sampler is personally responsible for the care and initiation of custody of the samples collected until they are transferred to the Sample Coordinator.

2. All bottles will be tagged with the sample numbers and locations. The date, time sampled, analyses to be performed. and sample collector's signature also will be entered on the tag.

3. Sample tags (Figure 7) will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a field logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

4. The Field Team Leader will review all field activities to determine whether proper custody procedures were followed during the field work, assess whether proper documentation was filled out, and decide if additional samples are required.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the base could reconstruct a particular situation without reliance on memory. As stated in the FSP, dedicated field logbooks will be kept by all field personnel.

Field logbooks will consist of bound, waterproof, field survey books with numbered pages. Logbooks will be assigned to field personnel, but will be stored securely in the Dames & Moore office located at 6310 Lamar Avenue, Overland Park, Kansas, when not in use, or in locked storage at the site. After project completion, these documents will be in the custody of the Project Manager. Each logbook will be identified by a project specific document number.

The title page of each logbook will contain the following:

1. Person to whom the logbook is assigned.
2. Logbook number/project specific document number.
3. Richards-Gebaur AFB, MO, F41624-94-D-8102-Delivery Order _____,
4. Starting date for the logbook.
5. Ending date for the logbook.

Entries in the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site and the purpose of their visit, and field sampling or investigation team personnel will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed and dated. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the sample, which includes direction and distance measurements from readily-definable site features (e.g., the corner of a building), shall be recorded. All sample locations will be surveyed by a Missouri-licensed surveyor. The number of the photographs taken of the sample station, if any, will also be noted.

Field samples and field QC samples will be collected following the sampling procedures documented in the FSP and at the frequency stated in this QAPP. The equipment used to collect samples will be recorded in the field logbook, along with the time/date of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection, and sample containers will be pre-labeled, where possible. Field duplicate and blank samples will receive an entirely separate sample identification number, and they will be noted under sample description as QC samples.

The Sampling Coordinator will be responsible for the completion of the Sample Logbook which provides details for all samples collected and shipped, including field sample identification number, sample description, sample depth, matrix, number of containers filled, analytes, date of collection/date of shipment, name of the sampler, sample container lot number, time and method of shipment, airbill number, location of the receiving facility, and PID results as needed.

5.1.3 Transfer of Custody and Shipment Procedures

The following procedures will be used when transferring custody of samples.

1. Samples will be accompanied by a properly completed chain of custody form. The sample numbers and locations will be listed on the chain of custody form (Figure 8). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the Sampling Coordinator, to the analytical laboratory, or to/from a secure storage area.

2. Samples will be properly packaged for shipment and dispatched to the analytical laboratories for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the container. The custody seals are covered with clear plastic tape. The container is strapped shut with strapping tape in at least two locations.

3. Whenever samples are split with AFCEE MDNR or the USEPA, a separate Sample Receipt will be prepared for those samples and marked to indicate with whom the samples are split. The person relinquishing the samples to the agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this will be noted in the "Received By" space.

4. All shipments will be accompanied by the Chain-of-Custody Record identifying the contents. The original record will accompany the shipment, and the gold copy will be retained by the sampler and returned to the Dames & Moore project office and placed in the project files. The laboratories will return the original copy of the custody form with the laboratory results.

5. The chain-of-custody document will be placed inside the shipping container in a sealed Ziploc® plastic bag.

6. If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. United

Parcel Service (UPS) or Federal Express will serve as the couriers for sample shipments; as such, copies of the standard UPS or Federal Express airbills will be maintained as part of the permanent documentation. The couriers will not sign the chain-of-custody form, as the coolers will be sealed with custody seals prior to pickup.

7. Samples requiring refrigeration will be promptly chilled with ice or "Blue Ice" (ice is preferred over "blue ice") and packaged in an insulated cooler; the volume of blue ice must be sufficient to maintain a temperature of approximately 4°C during transport to the analytical laboratory.

8. Only shipping containers which meet all applicable State and Federal standards for safe shipment will be used.

When a 4°C requirement for preserving the sample is indicated, the samples must be packed on ice or chemical refrigerant to keep them cool during collection and transportation. It is acknowledged that during transit it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. Sample shipping container (cooler) temperature must be checked when samples are received and must be recorded on the chain-of-custody forms. It is impossible to set acceptance temperature limits for the cooler temperature because of the complexity of the issue. When, in the judgment of the laboratory, the temperature of the samples upon receipt may have affected the stability of the analytes of interest, the problem must be discussed by the prime contractor with the AFCEE.

5.1.4 Field Documentation Responsibilities

It will be the responsibility of the Field Team Leader to secure all documents produced in the field (i.e., geologists' daily logs, lithologic and sampling logs, communications, etc.) at the end of each day's work. The Sampling Coordinator will be responsible for review of the documents for accuracy against the sample log.

The possession of all records will be documented; however, only the Field Team Leader and Project Manager or their designee may remove field data from the site for reduction and evaluation.

5.1.5 Sample Numbering Designation

A unique sample numbering system will be used to identify each sample for chemical or geotechnical analysis or field screening. In addition, this sample numbering system will be used to identify all trip blanks, field equipment blanks, field duplicates and additional sample volume designated for MS/MSD analyses. Each unique sample number will consist of the components described below.

The following 3-letter designation will be used to identify the Richards-Gebaur SC Delivery Order 0002 field samples:

RG2 = Richards-Gebaur SC Delivery Order 0002

Each sample location will be identified by an alpha-code corresponding to the sample type, followed by a 2- or 3-digit sample location number, as appropriate. The alpha codes are as follows:

MW = monitoring well
PZ = piezometer
BH = soil boring
RW = river water
SD = sediment
SS = soil sample

The following codes will be used for QC samples, and will be added as prefixes to the sample location:

TB = trip blank
FB = field equipment blank
FD = field duplicate

MS/MSD = matrix spike/matrix spike duplicate

Sample locations and their numbers are shown in Figure 3. (This figure will be developed to reflect sampling pertinent to specific DOs). Soil sampling depths will be designated by the depth of the bottom of the 2-foot sample interval (i.e., a soil sample from a depth of 8 to 10 feet would be given the suffix "10").

The following are examples of the sample numbering to be used during the project:

RG2 - MWP100A:	Richards-Gebaur SC Delivery Order 0002, ground water collected from monitoring well P100A
RG2 - PZ01:	Richards-Gebaur SC Delivery Order 0002, ground water collected from piezometer 01
RG2 - BHP100B-10':	Richards-Gebaur SC Delivery Order 0002, soil collected from boring P-100B at a depth interval of 8 to 10 feet
RG2 - SD24-10':	Richards-Gebaur SC Delivery Order 0002, sediment collected from location 24 in the river at a sediment depth interval of 8 to 10 feet below the water level
RG2 - FDSD24-10':	Richards-Gebaur SC Delivery Order 0002, field duplicate sediment collected from location 24 in the river at a sediment depth interval of 8 to 10 feet below the water level
RG2 - TB03:	Richards-Gebaur SC Delivery Order 0002, third trip blank collected and shipped with cooler containing water samples for volatile organic compound analysis
RG2 - MS/MSDMW-21:	Richards-Gebaur SC Delivery Order 0002, additional volume of water from monitoring well 21 for laboratory matrix spike/matrix spike duplicate.

5.2 LABORATORY CUSTODY PROCEDURES

The custody and documentation of the sample must be traceable and secure after it arrives at the laboratory. This section discusses the laboratory operations necessary to ensure sample and document integrity.

5.2.1 Chain-of-Custody Documentation

The trail of the sample's journey through the laboratory, from log-in to disposal, is documented by an unbroken written record chain-of-custody form, (COC) that accounts for the secure location of the sample at all times.

All samples analyzed by Chemron just be traceable to a person or secured area at any point in the analysis. This is required to prove sample integrity for legal purposes. It is accomplished by the use of a chain-or-custody system, which records the disposition of every sample at every point in the sampling/analysis procedure. Each sample received by Chemron must have an accompanying COC form giving all the information pertaining to the field sampling. It should be signed for the laboratory by the person first introducing the sample into the laboratory system. Whenever a sample/extract is removed from the refrigerator or other storage area, the COC form for that storage area must be filled out with the Laboratory ID, Date, purpose and person responsible. Likewise, when the sample/extract is returned to the storage area or sent to long-term storage, the same information is required.

5.2.1.1 Receipt and Log-In

The sample custodian has primary responsibility for entering incoming samples into the laboratory system. When received, all samples must be logged-in to the package log in the front office, before transferring them to the laboratory for opening. The sample custodian then inspects each sample container for signs of leakage, breakage, temperature, seal breakage, or any other sign of compromised sample integrity. This information is recorded on the COC form and Dames & Moore Project Manager will be notified if necessary. The sample is then assigned a laboratory ID number and entered into the lab computer system. Until preparative extraction, the sample is placed in the sample refrigerator, and noted on the log.

5.2.1.2 Aliquoting After TCLP

The TCLP technician will be responsible for aliquoting the leachate into separate containers for transferal to the analytical sections. The following will be the standard quantities needed for each analysis.

Semivolatiles	-	GC/MS	-	1 liter bottle
Volatiles	-	GC/MS	-	1 125 ml Tedlar bag
Pesticides	-	GC/ECD	-	1 liter bottle
Herbicides	-	GC/ECD	-	500 ml bottle
Metals	-	AA	-	200 ml bottle

All glassware cleaning protocols, storage conditions and holding times, applicable to each method, will be strictly observed. Extra portions of leachate are to be used for preparation of matrix spikes, or reanalyses (if properly handled).

5.2.1.3 Standard and Sample Labeling

All containers (including flasks, bottles, Tedlar bags, beakers, etc.) are carefully labeled as to the contents they contain. The minimum information required is:

a. Laboratory ID Number or Contents
b. Solvent Solutes (analytes, etc.)
c. Concentration (for standards)
d. Date prepared or filled
e. Analysts initials
f. Hazardous properties (if possible)
g. Dilution (if applicable)
h. Lot number (of all reagents used).

If all of the information does not fit on the label, it is recorded in a laboratory notebook and reference made on the label.

Labels are written with non-erasable/non-smearing ink and securely affixed to the container. For vials, or other small containers, information can be written in permanent marker directly on the container.

The analyst takes care that all container labels are maintained as stated above. Any problems or illegible labels are taken care of immediately.

5.2.1.4 Sample Storage

Once analysis is completed, the samples are segregated into Hazardous, Hon-Hazardous, and PCB contaminated storage areas. When the holding times are expired, the samples are disposed of as follows:

a.	PCB Liquids	-	Detox
b.	PCB Solids	-	Debris Drums
c.	Hazardous Samples	-	Return to customer
d.	Non-hazardous Samples	-	Return to customer or landfill

5.2.2 Document Control

The goal of the laboratory's document control system is to be able to supply to Dames & Moore documents relating to the analysis of the Richards-Gebaur SC samples. These documents include, but are not limited to: chain-of-custody forms, sample bottle lots used, sample tags, airbills, bench sheets, logbooks, telephone conversation records, out-of-control forms, QA and/or progress reports, corrective action forms, accompanying QC sample results, calibration records, worksheets with calculations, instrument printouts, and final result sheets. The criteria for an acceptable document control system is that the data and records are secure, retrievable and complete.

The following document control procedures assure that laboratory records are able to be assembled and stored, for efficient retrieval upon request.

1. All laboratory records will be stored in the secured laboratory area so they are not accessible to laboratory visitors or instrument service personnel.

2. Locked file cabinets will be utilized to store completed records, and a check out card system is used for removal of working project files or archived files.

3. All original laboratory forms and data will be included in the project file, when all data from the project is compiled.

4. All pre-printed laboratory forms and logbook pages must contain the signature of the person conducting the work and the date the work was performed.

5. The pages in bound and unbound logbooks must be sequentially numbered. No pages will be removed. Bound logbooks will also be assigned a unique number.

6. Proper error correction procedures must be followed. No information will be obliterated or rendered unreadable. The unused portions of documents will be crossed out with a "Z" and the person's initials and date entered.

7. All documents, notebooks and forms will be completed in ink.

8. Project files will be kept for six years after acceptance of the Richards-Gebaur SC, in accordance with the terms of the Delivery Order.

9. Any changes to the laboratory information management system must be documented.

10. All telephone conversations related to the Richards-Gebaur SC must be documented, and the original filed with the project file. All faxes, fax cover sheets and transmission reports also must be filed in the project file.

5.3 FINAL EVIDENCE FILES

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. Dames & Moore is the custodian of the evidence file and maintains the contents of evidence files for the Richards-Gebaur SC, including all relevant records, reports, logs, field notebooks, pictures,

subcontractor reports and data reviews in a secured, limited access area and under custody of Dames & Moore's Base Manager. Project records will be retained in accordance with the procedures described in the DO.

> ***Record Keeping.*** *The Contractor shall create and maintain in one location written and electronic records sufficient to recreate each sampling, analytical, testing and monitoring event. The Contractor shall make these records available to the government per DO SOW paragraph 3.2.7. The Contractor shall maintain records of, and derived from, all activities outlined in the appropriate portion of the QPP supporting the generation of these sampling and analysis records. The Contractor shall also retain written calculations using information obtained from sampling, analysis monitoring and testing activities, to include all raw data. All data entered into the Installation Restoration Program Information Management System (IRPIMS) data files and submitted by the Contractor shall correspond exactly with the data contained in the original laboratory reports and other documents associated with sampling and laboratory contractual tasks.*

The laboratory will maintain evidence files for the project analytical data generated by that laboratory for a minimum of six years after approval of the SC. At that time, Dames & Moore will be contacted by the laboratory to determine the final disposition of the information.

The final evidence file will include, at a minimum:

- Field logbooks.
- Field data and data deliverables.
- Photographs.
- Drawings.
- Soil boring logs.
- Laboratory data deliverables.
- Data validation reports.
- Data assessment reports.
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, airbills, etc.).
- Draft and final reports.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures, and the frequency at which these procedures will be performed, for both field and laboratory instruments.

6.1 FIELD INSTRUMENT CALIBRATION

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used during the field sampling will be examined to certify it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment programs are not overlooked, and all necessary repairs to equipment have been carried out. Backup equipment will be available.

Calibration of field instruments is governed by the specific operating procedures for the applicable field analysis method, and such procedures take precedence over the following general discussion. Operating procedures for all equipment to be used on the project are addressed in the FSP. Calibration of field instruments will be performed at the interval specified by the manufacturer or more frequently as conditions dictate. Figure 9 provides an example equipment calibration form.

6.1.1 Photovac MicroTip™ Photoionization Detector (PID)

The PID provides semi-quantitative concentration readings of the volatile compounds in a sample that have ionization potentials equal to or less than 10.6 eV. The PID will be calibrated each day in the field prior to the start of sampling activities, using 100 ppm isobutylene span gas, and monitored for proper response between each screening event. Peak instrument readings will be recorded on the soil boring logs, field logbooks, and on the sample container label for use by the laboratory. PID readings from collocated samples will be assumed to be similar to primary samples.

Periodic calibration is required to compensate for PID output changes due to inlet filter restriction, lamp window cleanliness, sample pump wear and other factors. Calibration procedures and frequency will be recorded in field logbooks. General procedures for calibration are described below:

1. Turn the unit on and wait until "Warming up now" message is replaced by "Ready" message.

2. Touch the CAL button. At the message "Connect zero gas then press ENTER" press ENTER. Usually clean outdoor air will be suitable as Zero Gas.

3. At the message "Span conc? ppm", enter 100. Then connect the 100 ppm isobutylene span gas (using a bag adapter) to the PID inlet. Press ENTER.

4. When the PID display reverts to normal ("Ready"), calibration is complete and the unit is ready for use.

6.1.2 Hanna 8424 Digital pH Meter

The Hanna 8424 is a portable, microprocessor-based pH meter. A new instrument must calibrated before use in pH measurement. Re-calibration will be necessary after the battery is replaced, or if the pH electrode or the ATC probe are replaced. The instrument can be calibrated with a pH 7.00 and either pH 4.01 or pH 10.00 standard technical buffers. (If you are measuring between 0 and 7 pH, use pH 7.00 and pH 4.01 buffers. If your measurements will be between 7 and 14 pH, use pH 7.00 and pH 10.00 buffers.)

Calibrations will be recorded in a field logbook along with the lot numbers of the buffers. General procedures for the pH meter are described below. NOTE: When using the ATC probe, the meter automatically compensates for changes in buffer temperature. Ideally, buffer solutions should be kept at 25° C for calibration. When not using the ATC probe, make sure that the temperature is set manually to reflect the temperature of the solution.

1. Place the electrode in a pH 7.00 buffer solution. Wait approximately 30 seconds for the sensor to stabilize, and then press CAL.

2. If the electrode recognizes the pH 7.00 solution, the exact value will appear on the display. If not, the symbol "E4" will be displayed - see "Error Code Guide" in the manufacturer's manual.

3. Wait 30 seconds and then push CON to accept the buffer value.

4. Take the electrode out of the pH 7.00 solution, rinse it with distilled water and dip it into the pH 4.01 or pH 10.00 solution. The display "E5" will disappear when the electrode is placed in the second buffer, and the value of the chosen buffer will then appear on the display. Wait 30 seconds and press CON. The instrument is now calibrated and will remain calibrated even after the unit is shut off.

6.1.3 Horiba U-10 Water Quality Checker

The Horiba U-10 Water Quality Checker is a portable instrument for the simultaneous multiparameter measurement of water quality. The instrument measures six different parameters of water quality: pH, conductivity, turbidity, dissolved oxygen, temperature, and salinity. The U-10 is compact enough to be held in one hand while taking measurements. Measurements are taken by immersing the probe into the water sample. The specific instructions on the calibration frequency, the acceptance criteria and the conditions that will require more frequent recalibration are presented in the Model U-10 Instrument Manual. The manual will be kept in the field during sampling to serve as the SOP for operation. The following information was excerpted from the Model U-10 Manual with permission from the manufacturer.

The U-10 Water Checker can be calibrated either manually or automatically. The four-parameter auto-calibration procedure is rapid and useful for periodic checks during normal operations. Manual calibration for each of the four parameters is more accurate but more time-consuming. Manual calibration is used for difficult measurements or where greater than normal precision is required. For this project, manual calibration will be performed at the start of the project, and will be supplemented with auto-calibration on a daily basis during sampling.

6.1.3.1 Auto-Calibration Procedure

The U-10 Water Checker is supplied with a bottle of standard phthalate pH solution and a calibration beaker for the purpose of auto-calibration. Fill the calibration beaker about two-thirds full with the standard solution. Note the fill line on the beaker. Fit the probe over the beaker. Note that the beaker is specially shaped to prevent the DO sensor from being immersed in the standard solution; this is because the DO auto-calibration is done using atmospheric air.

With the power on, press the MODE Key to put the unit into the MAINT mode. The lower cursor should be on the AUTO Sub-Mode; if it is not, use the MODE Key to move the lower cursor to AUTO.

With the lower cursor on AUTO, press the ENT Key. The readout will show CAL. Wait a moment, and the upper cursor will gradually move across the four auto–calibration parameters one–by–one: pH, COND, TURB, and DO. When the calibration is complete, the readout will briefly show END and then will switch to the MEAS mode. The upper cursor will blink while the auto–calibration is being made. When the auto–calibration has stabilized, the upper cursor will stop blinking. If you wish to abort the auto–calibration for any reason, press the CLR Key. The parameters auto–calibrated so far will be stored in the memory.

After the DO auto–calibration, if the unit does not switch to the MEAS mode as it should, and the readout shows either ER3 or ER4, an auto–calibration error has occurred. Parameters will blink where an error occurred. If this happens, re–do the auto–calibration. First, press the CLR Key to cancel the error code. Then press the ENT Key to re–start the auto–calibration. Restart the auto–calibration beginning with pH.

6.1.3.2 Manual (2-point) Calibration Procedures

The manufacturer recommends that for normal measurements, the four–parameter auto–calibration described above is sufficiently accurate. A two–point manual calibration of one or more of the four parameters is recommended either for high–accuracy measurements or especially when using the expanded readout mode. It is necessary if a new probe is being used for the first time. Parameters to be manually calibrated include pH, conductivity, turbidity, and dissolved oxygen.

pH Calibration

1. Zero Calibration

Wash the probe two to three times, using deionized or distilled water. Place the probe in a beaker of pH 7 standard solution, i.e., a neutral phosphate standard solution. With the power on, press the MODE Key to put the unit into the MAINT mode. Press the MODE Key again to move the lower cursor to ZERO. Use the SELECT Key to move the upper cursor to pH. When the readout has stabilized, use the UP/DOWN Keys to select the value of the pH 7 standard solution at the temperature of the sample. Refer to Table 2, page 25, of the Instrument Manual for pH values of standard solutions at various temperatures. Press the ENT Key to complete the zero calibration for pH.

2. Span calibration

Wash the probe two to three times in deionized or distilled water. Place the probe in a beaker of either pH 4 or pH 9 standard solution. Use the MODE Key to move the lower cursor to SPAN. As in the zero calibration, when the readout has stabilized, use the UP/DOWN Keys to select the value of the standard solution (i.e., either pH 4 or pH 9) at the temperature of the sample. Again, refer to Table 2 of the Manual for pH values of standard solutions at various temperatures. Press the ENT Key to complete the span calibration for pH.

Conductivity Calibration

The U–10 can measure conductivity in the range of 0–100 mS/cm. Depending on the sample concentration, however, the U–10 automatically selects the proper range out of its three possible ranges of 0–1 mS/cm, 1–10 mS/cm, and 10–100 mS/cm. If a manual calibration for Conductivity is being performed, the procedure must be done for each of the three ranges. However, since the zero point is common for all three ranges, only the three one–point span calibrations need be done separately.

1. Preparing the standard solution for COND span calibration

This procedure uses a potassium chloride standard solution. For greater accuracy, the solution should be freshly prepared each time. If it is necessary to use a stored solution, be sure it has been kept tightly capped in a polyethylene or hard glass bottle. The shelf life of this solution is six months; date–stamp the bottle for reference. Never use a KCl standard solution that has been stored for more than six months: the calibration accuracy may be adversely affected.

Use potassium chloride powder of the best quality commercially available. Dry the powder for 2 hours at 105 C, and cool it down in a desiccator. Weigh the appropriate amount of dried and cooled potassium chloride powder according to the table presented in the U–10 Manual, page 27. To prepare the standard solution, use a 1–liter volumetric flask. First, dissolve the KCl in a small amount of deionized or distilled water. Then fill the flask with deionized or distilled water to the 1–liter line. Finally, shake the solution to mix it thoroughly.

2. Zero calibration

Wash the probe two to three times, using deionized or distilled water and shake the probe to remove any water droplets from the COND electrode. Allow it to dry exposed to fresh air. Use the MODE Key to move the lower cursor to ZERO. Use the SELECT Key to move the upper cursor to COND. Use the UP/DOWN Keys to set the readout to 0.0. Press the ENT Key. This completes the zero calibration for COND.

3. Span calibration

Wash the probe two to three times using de–ionized or distilled water. Following this, wash it two to three times in the KCl standard solution you have prepared. Place the probe in a beaker of the KCl solution maintained at a temperature of 25 +5 C. Use the MODE Key to move the lower cursor to SPAN. After the readout stabilizes, similar to the pH calibration, use the UP/DOWN Keys to select the value of the KCl standard solution, referring to the KCl table. Press the ENT Key to complete the span calibration for this COND range. Repeat this procedure for the three ranges, using each of the three values of KCl standard solutions.

Turbidity Calibration

Wash the probe two to three times, using deionized or distilled water. For the span calibration, use a prepared span solution. For the turbidity zero calibration, use deionized or distilled water.

1. Preparing the standard solution for TURB span calibration

Weigh 5.0 g of hydrazine sulfate. Dissolve this in 400 ml of deionized or distilled water. Then weigh 50 g of hexamethylenetetramine, and dissolve it in 400 ml of deionized or distilled water. Mix these two solutions, add enough deionized or distilled water to make 1,000 ml, and stir the mixed solution thoroughly. Allow this solution to stand for 24 hours at a temperature of 25 + 3 C.

The turbidity of this solution is equivalent to 4,000 NTUs. The shelf–life of this solution is 6 months, i.e., this 4,000 NTU value will remain accurate for a maximum of 6 months.

Each time you carry out this calibration, it is necessary to dilute the 4,000–NTU standard solution to prepare an 800–NTU standard solution for calibration. To do this, place 50 ml of the 4,000–NTU solution into a 250–ml measuring flask; it is recommended that a rubber pipette aspirator be used to do this. Then add deionized or distilled water to the 250–ml line.

The standard solution used for the turbidity calibration will precipitate easily. Therefore, be sure to stir the solution thoroughly before use.

2. Zero calibration

Wash the probe thoroughly two to three times using deionized or distilled water. Shake off excess water droplets, and place the probe in a beaker of deionized or distilled water.

Use the MODE Key to move the lower cursor to ZERO. Use the SELECT Key to move the upper cursor to TURB. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys. Press the ENT Key to complete the zero calibration for TURB.

3. Span calibration

Wash the probe thoroughly, using deionized or distilled water, and shake off excess water droplets. Then place the probe in a beaker of the 800–NTU solution you have prepared for this purpose.

Stir this 800–NTU span standard solution thoroughly. Use the MODE Key to move the lower cursor to SPAN. After the readout has stabilized, (approximately 60 to 90 seconds), set the readout to "800" NTU, which is the value for this standard solution. Press the ENT Key to complete the span calibration for TURB.

Dissolved Oxygen Calibration

A zero standard solution is used for the DO zero calibration. An oxygen–saturated span solution is used for the DO span calibration.

1. Preparing the standard solution

Zero solution – Add approximately 50 g of sodium sulfite to 1,000 ml of water (either deionized water or tap water). Stir this mixture thoroughly until completely dissolved.

Span solution – Put 1 to 2 liters of water in a container (either deionized water or tap water). Use an air pump to bubble air through the solution until it is oxygen–saturated.

2. Zero calibration

Wash the probe two to three times in tap water, and place it in the zero standard solution. Use the MODE Key to move the lower cursor to ZERO. Use the SELECT Key to move the upper cursor to DO. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys. Press the ENT Key. This completes the zero calibration for DO.

3. Span calibration

Wash the probe two to three times in tap water, and put it in the span standard solution. Be sure the U–10 is set for fresh water readings. To do this, set the S.SET Sub–Mode to 0.0%. Then, use the MODE Key to move the lower cursor to SPAN. After the readout has stabilized, while slowly moving the probe up and down in the solution, set the readout value to the appropriate DO value for the temperature of this solution. For DO values at various temperatures, refer to the table on page 34 of the U–10 Manual. Press the ENT Key to complete the span calibration for DO.

Temperature Calibration

Temperature measurements are carried out using alcohol thermometers to avoid possible site contamination by mercury if the thermometer were to break. Thermometers will be inspected when purchased to ensure the alcohol is intact. If a mercury thermometer must be used, it will be inspected to ensure there is no mercury separation. Thermometers will be checked biannually for calibration against an NIST reference thermometer at the ice point. Tolerance must be within ±0.5°C of 0°C or the thermometer will be discarded.

6.2 LABORATORY INSTRUMENT CALIBRATION

In general, calibration procedures for specific laboratory instruments will consist of initial calibration (3 or 5–points), initial calibration verification and continuing calibration verification. Descriptions of the calibration procedures for specific laboratory instruments, including frequency, acceptance criteria and conditions that will require re–calibration, are stated in the SOPs in the Appendix. In all cases the initial calibration will be verified using an independently–prepared calibration verification solution.

The laboratory maintains a sample logbook for each instrument which contains the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

7.0 ANALYTICAL PROCEDURES

Samples collected during field sampling activities for the Richards–Gebaur AFB SC will be analyzed by Chemron for the analytes listed in Table 5.

7.1 FIELD ANALYTICAL PROCEDURES

The procedures for the field measurements for total volatile organics (using a PID), pH, conductivity, turbidity, water level, and temperature are described in the field SOPs appended to the FSP.

7.2 LABORATORY ANALYTICAL PROCEDURES

The laboratory will implement the project–required SOPs as described in Appendix A. Chemron SOPs for sample preparation, cleanup and analysis are based on SW–846, Third Edition, Revision I, 1987, and EPA–600/4–79/020, *Methods for Chemical Analysis of Water and Waste Water*. These SOPs provide sufficient details and are specific to this SC.

Prior to taking an aliquot of soil/sediment for chemical analysis, the sample container contents will be examined for foreign objects (e.g., cigarette butts, paper, broken glass, debris) and stones; these items will be excluded from the subsample taken for analysis, and a notation will be made in the extraction log sheet of the presence of these items in the sample provided by the field staff.

Table 14 summarizes the analyte groups of interest and EPA reference methods to be evaluated in this investigation. The specific analytes to be determined by each group are presented in Table 3. The specific laboratory SOPs to be used in this investigation are included in Appendix A.

No significant deviations from stated EPA reference methods are included in the laboratory–specific SOPs used by Chemron Laboratories.

A complete listing of project target compounds and current laboratory reporting limits for each analyte are listed in Table 3. Practical quantitation limits have been experimentally derived.

Samples exhibiting large organic non–target peaks will be diluted only if the peaks interfere with surrogate recovery calculation or target compound quantitation. No reanalysis will be done if surrogates or MS/MSD analytes are diluted out. Similarly, if dilutions are needed to be able to physically aspirate the sample into the ICP/MS, GFAA, or ICAP instruments, exceeding the MS recovery or RPD limits will not be sufficient triggers for redigestion/reanalysis determined by using the method found in FR vol. 49, no. 209, page 198–199.

All dilutions should keep the response of major constituents in the upper half of the linear range of the curve. Target reporting limits, as listed in Table 3, will not be achievable in samples requiring dilution.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 FIELD QUALITY CONTROL CHECKS

During field activities, Dames & Moore will collect soil, ground water, surface water, and sediment samples for laboratory analysis. Table 1 provides a summary of the sampling and analysis effort of the project. Table 4 provides a listing of the QC samples that will be collected in the field; the field QC samples will consist of trip blanks, field equipment blanks, and field duplicates.

QC checks will be used to assess precision and bias of field measurements and will consist of repeating a measurement on selected samples. QC checks will be conducted at a frequency of 5 percent; measurements on the twentieth sample will be repeated. QC procedures for pH, conductivity, turbidity, and dissolved oxygen measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard, and by calibrating the Horiba U–10 instrument. The thermistor in the Horiba U–10 water checker (used to measure the temperature in aqueous samples) and the thermometer used to measure temperature in sediments and soil samples will be compared to an NIST traceable thermometer (or equivalent). Instrument calibration for the Horiba U–10 is discussed in Section 6.1.3.1. The acceptance criteria for field measurements are presented in Table 6 and Table 9 in Section 3.1.1. (precision) and Section 3.2.2 (accuracy).

8.2 LABORATORY QUALITY CONTROL CHECKS

There are two types of QA used by Chemron to ensure analytical data of known quality: program QA and analytical method QC.

Written QA programs which provide rules and guidelines to ensure the reliability and validity of work conducted at the laboratory have been prepared. Compliance with their QA programs is coordinated and monitored by the laboratory's Quality Assurance Officer (QAO), who is independent of the operating departments.

The stated objectives of the laboratory QA program are to:

1. Ensure that all procedures are documented, including any changes in administrative and/or technical procedures.

2. Ensure that all analytical procedures are conducted according to sound scientific principles and have been validated.

3. Monitor the performance of the laboratory by a systematic inspection program and provide for corrective action as necessary.

4. Ensure that all data are properly recorded and archived.

All laboratory procedures are documented in writing and are controlled by the Site Quality Assurance Manager. Internal quality control procedures for analytical services will be conducted by the laboratory in accordance with its standard operating procedures and the individual method requirements in a manner consistent with their QA programs. These specifications include the types of audits required (sample spikes, surrogate spikes, reference samples, controls, blanks), the frequency of each audit, the compounds to be used for sample spikes and surrogate spikes, and the quality control acceptance criteria for these audits.

The laboratory will document, in each data package provided, that both initial and ongoing instrument and critical analytical QC functions (such as lab method blanks and LCS recovery) have been met. Any samples analyzed in non– conformance with the critical QC criteria will be reanalyzed by the laboratory.

8.2.1 Summary of Laboratory Quality Control

QC protocols for analytical analyses include the following items.

Note: A group of 20 or fewer samples that are similar in composition (matrix) prepared (e.g., extracted or digested) together are defined as a "batch".

- A minimum of one method blank is analyzed per sample batch to detect contamination during preparation and/or analysis.

- One laboratory control sample (LCS) consisting of target analytes spiked into a blank matrix and analyzed per sample batch to determine accuracy and precision.

- One matrix spike and one matrix spike duplicate for organics analyses and one matrix spike and one matrix duplicate for inorganic analyses will be analyzed for every batch to determine the affect of the matrix on the method performed.

- Internal and surrogate standards will be added where appropriate to quantitate results, determine recoveries, and to account for sample–to–sample variation.

- Calibration of instrumentation will be performed according to the appropriate EPA methods.

The laboratory shall maintain records sufficient to recreate each analytical event conducted pursuant to the SOW. At a minimum, the records shall contain the following:

- Chain–of–custody forms

- Initial and continuous calibration records including standards preparation traceable to the original material and lot number.

- Instrument tuning records (as applicable).

- Method blank analyses.

- Internal standard results.

- Surrogate spiking and results (as required).

- Spike (and spike duplicate) records and results.

- Laboratory records.
- Raw data, including instrument printouts, bench work sheets, and/or chromatogram with compound identification and quantitation reports.
- Corrective action reports (see Section 2.2.6)
- Other method and project–required PC samples and results.

8.2.2 Specific QC Assignments by Sample Group

Specific laboratory QC samples that will be analyzed per sample group are as follows:

LCS = Laboratory Control Sample
MS = Matrix Spike
SD = Matrix Spike Duplicate
DU = Matrix Duplicate
MB = Method Blank [includes Single Control Sample (SCS) for Organics]

Organics: Per Batch – LCS, MS, SD, MB

Inorganics: Per Batch – LCS, MS, DU, MB when appropriate

Notes:

1. It is the responsibility of Dames & Moore to collect sufficient sample volume and designate MS/MSD analyses on the chain–of–custody.
2. Laboratory accuracy is determined through the results of the LCS/SCS. Sample matrix accuracy and precision are determined through the results of the MS/MSD.

In the analyses of samples in a preparation batch if the recoveries of analytes in the LCS are within the control limits, then the precision is also within limits.

Refer to the submitted Chemron SOPs (Appendix A) for a description of the specific QC requirements and the QC criteria.

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available, as discussed in Section 8.2.7. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary. Additional information on the Chemron QA program is detailed in the following sections.

8.2.3 Quality Assurance Objectives

Quality assurance objectives can be expressed in terms of precision, accuracy, representativeness, comparability, and completeness. Appendix A to the QAPP lists quantitative data quality objectives (precision, accuracy, and completeness) for the project–specific parameters.

Adherence to the DQOs will be measured quantitatively by comparing the results of the laboratory control samples (LCS) to control limits. A LCS consists of a standard control matrix which is spiked with a group of target compounds representative of the method analytes. For work on this DO, the LCS is a method blank spiked with known concentrations of all analytes of interest. One LCS is analyzed for every batch processed by a method.

As an additional measure of laboratory performance with each sample batch, a single control sample (SCS) is processed. An SCS consists of a control matrix that is spiked with surrogate compounds appropriate to the method being used. In cases where no surrogate is available, (e.g., metals or wet chemistry) the LCS serves as the control sample. An SCS is prepared for each sample batch.

Accuracy is defined as the degree of agreement between an observed value and an accepted reference value. The LCS is used to monitor the accuracy of the analytical method on an ongoing basis, independent of matrix effects. The LCS is monitored for accuracy (average

percent recovery) of each analyte in the LCS. Section 12 defines the calculation of Data Quality Indicators.

Percent completeness is defined as the number of valid data points obtained divided by the number of data points attempted. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for all of the analytical protocols. Less obvious is whether the data are sufficient to achieve the goals of the project; all data will be reviewed to determine if the data base is sufficient to accomplish the goals of the investigation.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. The FSP and QAPP have been designed to ensure the samples collected during the field investigation will be obtained, handled, shipped and analyzed in accordance with the requirements of the USEPA and AFCEE.

8.2.4 Control Limits

Control limits are generated for the standard control matrix based upon historical data where available. Control limits for accuracy and precision are subject to periodic updating, generally on an annual basis. The control limits used will be those in effect at the time the samples are analyzed and may be different from those listed in this document due to the periodic updating of these limits. Control limits listed in the SOPs represent the present control limits for Chemron. Guidance control limits for sample matrices are derived from reference methods (where available).

8.2.5 Laboratory Control Samples and Quality Assurance Objectives

Accuracy is assessed by the laboratory by comparing the results of the LCS to the control limits. Accuracy is expressed as the average percent recovery of the LCS.

For all tests, if the LCS is out of control limits, all samples which are associated with the unacceptable LCS are re–prepared and/or reanalyzed. Occasionally it is apparent that although the LCS is out of control, the samples associated with this LCS are unaffected and within all other QC criteria and the data is acceptable for its intended use. In these cases, the laboratory

would seek approval from Dames & Moore regarding the acceptability of the data and, if appropriate, may report the data with a narrative. All decisions such as this would be fully documented and technically supported in the narrative.

8.2.6 Single Control Samples and Quality Assurance Objectives

Recovery data generated from the SCS are compared to control limits that have been established for each of the compounds being monitored. Analytical data that are generated with an SCS which falls within the control limits are judged to be in control. Data that are generated with an SCS which falls outside of acceptance criteria are considered suspect and corrective action must be performed. The associated SCS data are reported with each set of sample results to enable a quality assessment of the data.

8.2.7 Matrix Specific QC

Matrix specific QC samples are analyzed as designated on the chain of custody. The performance of matrix specific analyses for aqueous matrices requires additional sample volume which must be collected and submitted at the same time as the original routine sample. Triple the volume will be collected for samples requiring MS/MSD.

Matrix spike/matrix spike duplicates will be collected at a frequency of one pair of MS/MSDs for each sample type per batch of twenty samples.

For organics analysis, the percent recovery and relative percent difference (RPD) of the matrix spike/ matrix spike duplicate (MS/MSD) will be calculated. For inorganic and other water quality analyses, the MS percent recovery and matrix laboratory duplicate RPD will be calculated. This allows for demonstration of the effect of the matrix on the method performed. Re–extraction and reanalysis decisions are made based on the LCS, method blanks, and QC requirements (e.g., surrogate recoveries) of the methods.

For all tests, if matrix spike recoveries exceed control limits and the batch laboratory QC samples (LCS, SCS) are within limits, a sample specific matrix effect is indicated. However, obvious problems with preparation and analysis, which can lead to poor recoveries, must be ruled out prior to attributing low recoveries to matrix effects. If no problems are identified, the

low recoveries will be noted in the report narrative and the effected data should be considered an "estimated concentration."

8.2.8 Surrogates

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of analysis. Results are reported in terms of percent recovery. LCS and SCS control limits which are generated by spiking a clean matrix are not used for judging surrogate recoveries in individual samples. Limits to which surrogate recoveries are compared are presented in Table 13, Section 3.2.3.

8.2.9 Method Blanks

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

As part of a standard QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, clean–up, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

Ideally, the concentration of target analytes in the blank should be below the Practical Quantitation Limit for that analyte. In practice, however, some common laboratory solvents and metals are difficult to eliminate to the parts–per–billion levels commonly reported in environmental analyses. Therefore, criteria for determining blank acceptability must be based on consideration of the analytical techniques used, analytes reported, and reporting limits required.

For organic analyses, the concentration of target analytes in the blank must be below the reporting limit for that analyte in order for the blank to be considered acceptable. An exception is made for common laboratory contaminants (methylene chloride, acetone, 2–butanone, and

phthalate esters) which may be present in the blank at up to three times the reporting limit and still be considered acceptable. This policy has been established to reflect the fact that these compounds are frequently found at low levels in method blanks due to the materials used in the collection, preparation, and analysis of samples for organic parameters.

For metals and wet chemistry analyses, where the reporting limits are typically near the Instrument Detection Limit (IDL), the policy is that the concentration of the target analytes in the blank must be below two times the reporting limit. If the blank value lies between the reporting limit and two times the reporting limit, the analyte in the associated samples are flagged to indicate contamination was present in the blank. A blank containing an analyte(s) above two times the reporting limit is considered unacceptable unless the lowest concentration of the analyte in the associated samples is at least ten times the blank concentration or the concentration of the analyte in all samples associated with the blank is below the reporting limit.

In addition, for wet chemistry tests, the method SOP directs how the blank is treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a prep blank is also analyzed to determine the extent of contamination or background interference. Blanks have no application or significance for some wet chemistry parameters (e.g., pH).

If the blank does not meet acceptance criteria, the source of contamination must be investigated and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank, and/or re–preparation and reanalysis of the blank and all associated samples.

For organic and metals analyses, and selected wet chemistry tests, method blank results are reported with each set of sample results. Sample results will <u>not</u> be corrected for blank contamination unless required by the analytical method. Occasionally, due to limited sample volume or other constraints, the laboratory reports data associated with an unacceptable blank. In these cases, the actual observed value is reported in the method blank and all sample results are flagged to indicate contamination was present in the associated method blank.

8.3 Special Quality Control for Gas Chromatography and ICP Analyses

- For gas chromatography (GC) methods, analyte retention times and retention time windows shall be established using the procedure in section 7.5 of method SW8000 and shall be used to ensure accurate identification of peaks.

- For GC methods, confirmation of analytes (except multi–response analytes) present in concentrations at or greater than the PQL is required. Confirmation may be accomplished either by quantitative second–column GC or by quantitative GC/mass spectroscopy (MS), provided the PQLs are equivalent. Data from both the initial analysis and the confirmation shall be reported. The result that Chemron considers to be the most reliable will be identified by Chemron. This result will not be obtained by averaging the two results.

- An inductively coupled plasma (ICP) spectrometer interference check standard (ICS) must be analyzed at the beginning and end of all analytical sequences, as specified in method SW6010, to verify interelement correction factors. If the ICS fails to meet acceptance criteria, corrective actions must be performed. When the quantitation of the affected analyte is critical to the project, reanalysis by Graphite Furnace Atomic Absorption (GFAA) must be carried out after discussions with the TC.

- Analyte concentrations may be determined with either calibration curves or response factors as defined in the methods. When using response factors to determine analyte concentrations, the average response factor from the initial calibrations, the average response factor from the initial calibration will be used, except in GC/MS methods. GC/MS quantitation shall be based on the response factor from the daily continuing calibration unless samples are analyzed in the sam sequence as the initial calibration. The continuing calibration shall be used for the sole purpose of verifying the initial calibration stability, and under no circumstances shall the laboratory update the response factors for the initial calibration to include subsequent continuing calibrations.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

This task describes the data reduction, validation and reporting for the laboratory operations, field activities and the ultimate classification of the data.

9.1 DATA REDUCTION AND VALIDATION

9.1.1 Field Data Reduction and Validation Procedures

All field measurements will be direct reading, and field reduction procedures will be minimal in scope.

The use of the Horiba U–10 water quality checker, thermometers, and PID meters will generate measurements directly read from the meters following calibration per manufacturer's recommendations as described in Section 6.0 of this QAPP. The data will be written directly into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed, and dated by the field member, and corrected in the space adjacent to the original (erroneous) entry.

After completing a sampling program, the field data package (field logs, calibration records, chain–of–custody forms, etc.) will be reviewed by the Field Team Leader and then the DO Manager for completeness and accuracy. Items to be included in field data validation are listed below:

1. A review of completeness of field data contained on field water and soil/sediment sampling logs

2. Verification that field blanks, trip blanks, and field duplicates were properly prepared, identified, and collected at the required frequency.

3. An on–site audit covering field analyses for equipment calibration and condition, and an assessment of the accuracy and precision of the field test data and measurements.

4. Verification that replicate field measurements were conducted at a frequency of 10 percent.

5. A review of chain–of–custody forms for proper completion, dates, signatures of field personnel and the Sampling Coordinator.

6. Review of the Sample Log for completeness and consistency with chain–of–custody paperwork and copies of airbills.

9.1.2 Laboratory Data Reduction Procedures

Chemron will perform data reduction and validation under the direction of its laboratory QAO. The laboratory QAO is responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other notations which would caution the data user of possible unreliability.

All analytical data generated are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and review.

Before the data report is released to AFCEE, the Dames & Moore QA Manager will review the report to ensure that the data meet the overall objectives of this QAPP.

9.1.3 Dames & Moore Data Reduction and Validation Procedures

The Dames & Moore Project Manager will review all chemical and geotechnical data received from the analytical laboratories prior to its inclusion in the SC Report to verify that it reasonably reflects known or expected conditions. The raw data collected from the project sampling tasks and used in project reports will be appropriately identified and included in a separate appendix of the SC Report. When test data have been reduced, the method of reduction will be described. Compound concentrations detected in field, trip, or method blanks will not be subtracted from other sample results.

After validation of the field data package to assess if samples were collected in accordance with this QAPP, validation of all the chemical analytical data packages will be

performed by Dames & Moore for all DQO Level 3 analyses. These data validation activities will typically consist of data review, data evaluation, data flagging, and data interpretation.

The review of laboratory data shall focus on the following subjects during validation:

- Chain–of–custody forms
- Holding times
- Method calibration limits
- Laboratory verification of quantitation limits
- Preparatory batch control records
- Corrective actions
- Formulas used for analyte quantitation
- Examples of analyte quantitation
- Completeness of data

The verification of quantitation limits and the establishment of control limits shall be confirmed.

Method validation is a continuous process and the reviewer shall ensure that control charts and statistical calculations have been updated to include recent data. Any control limits outside the acceptable range specified in the analytical methods shall be identified. Any trends or problems with the data shall be noted and evaluated in the TR. Any quantitation limits that exceed those in the SAP or this handbook shall be identified. The absence of records supporting the establishment of control criteria shall also be noted in the TR.

The results of preparation batch QC and calibration check samples shall be compared to SAP–specified acceptance criteria. Data not within control limits require corrective action. The reviewer will check that corrective action reports and the results of reanalysis are available. Similarly, sample holding times and preservations shall be compared to those required. If holding times were exceeded, evidence of resampling and analysis with the proper holding time or written variance from the AFCEE shall be noted. Samples associated with out–of–control QC data shall be identified in the TR, and an assessment of the utility of such analytical results shall be recorded. Corrective action reports shall be referenced in this assessment.

Method calibrations and continuing calibration verifications shall be reviewed to assure conformance to acceptance criteria and completeness of records. The review shall also ascertain that the calibration events can be recreated and that no project samples were analyzed when the instrument was not in proper tune or calibration. Quantitation reports shall be reviewed to assure correctness and completeness of calculated results. The formulas used and sample calculations shall be provided.

The check of laboratory data completeness shall ensure that all samples and analyses required by the SAP have been processed; complete records exist for each analysis and the associated QC samples; and the procedures specified in the WP, SAP, and SOPs have been implemented. The results of the completeness check will be documented in the TR in a tabular form.

9.1.3.1 Data Validation Reporting

Dames & Moore will prepare a data validation report for every sample delivery group received; that is, 20 percent of the DQO Level 3 data shall be validated. If inconsistencies are found 40 percent of the DQO level 3 data will be validated until data is clear or all are validated. The data validation report will be based on the results of the data validation process. As a minimum, every data validation report will contain the following information:

- Laboratory name
- Site name
- Sample number
- Sample results

- Data qualifiers
- Overall data assessment
- Explanation of action taken
- Comments

The data usability qualifiers and overall data assessment nomenclature and assignment procedures which will be used are a simplified versions of the EPA's. An example of the data usability qualifiers are:

V Data are valid
A Data are acceptable, but qualified due to problems
R Data are rejected
X Problems, but do not affect data

The data quality flags assigned will be:

R Code: Data flagged with an "R" has not met the required analytical QA requirements. This data is unusable even if field QC is acceptable.

J Code: Data flagged with a "J" has not met some of the analytical QA requirements; however, the problem was not of sufficient magnitude to warrant classifying the data as unusable. Data in this category is qualitative (estimated) provided the field data meets all criteria and the sample is valid.

U Code: The material was analyzed for, but was not detected. The associated numerical value is the sample quantification limit.

UJ Code: The material was analyzed for, but was not detected. The sample quantification limit is an estimated value.

B Code: The analyte was identified in the laboratory or field blank.

9.1.3.2 Classification of Data

Following the validation of the field data and the analytical data, a classification of the data for "use" will be performed. The data will be classified into one of three classes: Unusable, Class A (qualitative), or Class B (qualitative and quantitative).

Unusable Data: These are data that fail analytical quality control criteria and/or proper field and laboratory documentation is not obtainable.

Estimated Data: These are data (Class A) that do not meet the criteria for quantitative use. These data may be considered only estimated or qualitative. Data of this type may be used to evaluate presence or absence of chemical compounds and to help design additional sampling and analysis programs. These data may not be used for designing remediation or treatment systems.

Usable Data: These are data that meet all the requirements for documentation, qualitative use, and quantitative use. Data of this class (Class B) may be used for any purpose.

Failure to meet any criteria in Class A is required to be adequately explained, or the data for a given sample or sample matrix are classified as unusable. It should be noted that analytical data that were given "J" or "U" flags must be found to be acceptable under Class A (qualitative) criteria in order to be usable as qualitative data. These data, however, may not be considered for classification as Class B (quantitative) category. Data that were found to be analytically valid and passed all criteria for Class A may be considered for classification as Class B data.

As with Class A, failure to meet any criteria in Class B must be adequately explained or the data for a given sample or sample matrix may not be classified beyond Class A. Only data meeting all field and analytical data validation requirements and all Class A and Class B criteria may be classified as Class B data and used for qualitative and quantitative purposes.

Class A and Class B data will both be considered acceptable for the purposes of calculating completeness.

9.2 DATA REPORTING

9.2.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of boring logs, well completion forms, field logs, and daily reporting summaries containing tabulated results of measurements made in the field. Documentation of field calibration activities is also included in the TR. Field and laboratory data will also be entered and validated on the IRPIMS data base via CDLT for submission to AFCEE.

9.2.2 Laboratory Data Reporting

9.2.2.1 Analytical Reporting

The data will be reported in the same chronological order in which it is analyzed along with its associated QC data. The following information will be provided in each analytical data package submitted:

1. Cover sheet listing the samples included in the report and a case narrative describing problems encountered in analysis.

2. Tabulated results of inorganic and organic compounds identified and quantified.

3. Analytical results for QC sample spikes, sample duplicates, initial and continuous calibration verifications of standards and blanks, standards procedural blanks, laboratory control samples and ICP interference check samples.

4. Raw data system printouts (or legible photocopies) identifying the date of analyses, analyst, parameters determined, calibration curve, calibration verifications, method blanks, sample extraction and any dilutions, sample duplicates, spikes and control samples.

Data packages will include matrix spikes, matrix spike duplicates, surrogate spike recoveries, chromatogram, GC/MS spectra, and computer printouts. Specific data reduction and reporting procedures are provided in the laboratory's SOPs in Appendix A.

In general, reports will contain the following items.

- General Discussion — Descriptions of samples types, tests performed, problems encountered, and general comments are given.

- Analytical Data — Data are reported by sample by test and are not blank–corrected. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The reporting limit for each analyte is also given.

- QC Information — Analytical results for laboratory blanks are reported where applicable. In addition, the results (average percent recovery and relative percent difference) of the LCSs analyzed with the project are listed. Control limits are reported.

- Methodology — References for analytical methodologies used are cited.

9.2.2.2 Soils Laboratory

Dames & Moore's Soils Laboratory data reduction initially will be reviewed by the Dames & Moore Laboratory Manager. Validation will consist of data review by the Dames & Moore Project Manager or his designee.

9.3 PROJECT FILES

Project files will be created for each project handled within the Chemron laboratory, and will contain all documents associated with the project. This includes correspondence from the client, chain–of–custody records, raw data, copies of laboratory notebook entries pertaining to the project, and a copy of the final report. When a project is complete, all records will be passed to the document custodian who will put the files into the document archive. All files will

be secured in limited access areas and are signed in and out of the area. Raw data and all pertinent records will be retained for a minimum of six years after the termination of the Work Order. At that time the Dames & Moore Project Manager will be contacted to determine the final disposition of the files.

All hard copy analytical data deliverables will be generated at the time of analysis and be made available to AFCEE/ERB.

10.0 PERFORMANCE AND SYSTEM AUDITS

10.1 PERFORMANCE AND SYSTEMS AUDITS

Audits are planned and documented evaluations of project operations that determine the adequacy and effectiveness of, as well as compliance with, the project plans. Performance and systems audits of both field and laboratory activities will be conducted to verify that field sampling and chemical analyses are performed in accordance with the FSP and QAPP.

Performance audits verify the laboratory's ability to correctly identify and quantitate contaminants of concern in blind samples and as such, assess the accuracy of the measurement system. The analysis of any USEPA provided project–specific performance evaluation (PE) samples and laboratory participation in scheduled interlaboratory studies that might occur during the time of project sample analysis will be included as part of the performance audit.

The objectives of performance audits are:

- Ensure the QAPP developed for this project is being implemented;
- Assess the effectiveness of the QAPP; and
- Verify corrective actions are made in response to identified deviations.

Systems audits evaluate the entire process of generating the environmental measurement from sample collection through entry of the analytical value into the project database. Systems audits are initially performed prior to, or shortly after systems are operational, and then on a schedule during the project lifetime.

The objectives of systems audits are:

- Ensure that project documents (QAPP, FSP) are in use and understood;
- Verify that required documentation is accurate and complete;
- Assess effectiveness of communication and resolution of QA/QC deviations; and
- Verify effectiveness of corrective actions.

Performance and systems audits will be accomplished by two independent sources: internal and external assessors.

10.1.1 Internal Field Audits

Field audits will be conducted by the Dames & Moore Site Quality Assurance Manager. The Dames & Moore Field Team Leader will be responsible for correcting all deficiencies cited as deviations from the FSP and QAPP. The Dames & Moore Project Manager will be responsible for ensuring that corrective actions are complete and effective.

10.1.2 Internal Field Audit Frequency

An initial field systems audit will be conducted within the first week of initiation of field activities. A follow–up report based on a checklist will be used prior to the completion of the field sampling activities to verify that corrective actions are complete and effective.

10.1.3 Internal Field Audit Procedures

The primary purpose of the field systems audit is to verify that the field team is complying with the DQOs and properly documenting field activities. Examination of field sampling records, field instrument calibration and use logs, field custody procedures, sample container storage and labelling, packaging and shipping procedures and observation of sample collection and handling will be included. All field systems audits will consist of the following:

1. Pre–field mobilization preparation to familiarize the field team with the specific forms, field equipment calibration protocols, logs and instrument records to be kept during field activities, and their importance. This will be done by the Dames & Moore Field Team Leader and Project Manger with input from the Site Quality Assurance Manager.

2. Inspection of field records, with identification of documentation gaps, inconsistencies, and deviations from QAPP and FSP while on site using the checklist presented in Figure 10.

3. Observation of field sampling with immediate notification of Field Team Leader of deviations from the FSP.

4. Discussing the audit findings with the Field Team Leader at the site, and notification of the audit findings to the Project Manager.

5. Post–audit activities including preparation of an audit report detailing deviations, nonconformances, and corrective action recommendation. The Project Manager is then responsible to reply with corrective actions to be taken and the schedule of implementation.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

10.2.1 Dames & Moore Laboratory Audit Responsibilities

An internal audit of Chemron will be conducted for this project by the Dames & Moore Project Manager or the Dames & Moore Project Site Quality Assurance Manager using the checklist presented in Figure 11 developed by the Project Site Quality Assurance Manager.

The laboratory's Site Quality Assurance Manager will be responsible for implementing corrective actions to respond to deficiencies discovered by the audit. The Dames & Moore Project Manager/Site Quality Assurance Manager will be responsible for ensuring that corrective actions have been completed and are effective.

10.2.1.1 Dames & Moore Laboratory Audit Frequency

A systems audit Chemron will be done by Dames & Moore at least once prior to or during sample analysis (see Figure 12). A performance audit will be done in accordance with the laboratory's QA program. The accuracy of project analytes will be assessed at least once during the course of sample analysis, either by commercially purchased or internally prepared blind solutions.

10.2.1.2 Dames & Moore Laboratory Audit Procedures

The audited laboratory's Site Quality Assurance Manager and Operations Manger will be notified by the Dames & Moore Project Manager of an impending audit at least 2 weeks prior to the audit. This notification will be in writing and will include the scope and schedule of the audit and the name of the auditor. The audit checklist to be used will also be sent to enable the laboratory to have the supporting documentation identified readily available. The scope of the audit will include: personnel qualifications, availability, QA review process and documentation, sample reception and tracking, standards and reagent preparation/validation, lab method compliance with EPA reference methods, data entry, and report generation.

At the conclusion of the audit, a debriefing will be held to relay audit findings and clear up any misinformation. Deficiencies requiring corrective action will be stated and documented while on site in a brief summary. This summary will then be signed by the laboratory QAO and Operations Manager. If, during preparation of the final audit report, the Dames & Moore Site Quality Assurance Manager discovers additional deficiencies not cited during the debriefing, the laboratory QAO will be contacted and the items will be discussed prior to adding them to the audit report.

The audit report will be prepared within 7 working days of the audit by the Dames & Moore Site Quality Assurance Manager, and will be sent to the Dames & Moore Delivery Order Manager. The Dames & Moore Delivery Order Manager is then responsible for sending the audit report to the laboratory. The audit report will include the following:

1. Documents used in auditing (laboratory's own QA Manual, project QAPP).

2. Persons contacted during the audit.

3. Audit result summary presented at the debriefing.

4. Evaluation statements regarding the effectiveness of the laboratory QA program elements audited.

5. Detailed findings and program deficiencies to allow the development of appropriate corrective action.

6. Recommendations for correcting deficiencies or improving the QA program.

7. Notation of Good Laboratory Practices (GLP) needed/observed and non–imminent health and safety issues.

The laboratory will respond to the deficiencies in written form within 10 working days of receipt of the audit report. The response will clearly state the corrective action to be taken for each finding and deficiency, including action to prevent recurrence, and the date by which corrective action will be completed. If corrective action has already been completed, supporting documentation will be attached.

10.2.2 Internal Audits

Chemron is subjected to periodic systems audits by the QA department. These audits are intended to serve two purposes:

1. To ensure that laboratories are complying with the procedures defined in laboratory SOPs, QAPPs, and contracts.

2. To determine any sample flow or analytical problems. The frequency of the audits will be increased if any problems are suspected.

10.2.3 External Laboratory Audits

Formal lab audits may include (but are not limited to) review of laboratory analytical procedures, laboratory on–site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

External audits may be conducted as directed by AFCEE/ERB.

Additional external lab audits may be conducted by USEPA Region VII at any time before, during, or after the project's initiation. These audits may or may not be announced, and will be conducted at the discretion of the USEPA.

11.0 PREVENTIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTIVE MAINTENANCE

The field equipment for this project includes an electronic water level indicator, thermometers, a Horiba U–10 water quality checker, and a PID meter. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Equipment will be kept clean and securely stored to minimize maintenance problems. Procedures for use of the field equipment are provided in the Field Sampling Plan.

Field equipment will be checked and calibrated daily before use. Calibrations and calibration checks will be documented on the Field Meter/Calibration Log Sheets. An example of instrument calibration log is provided in Figure 12. Critical spare parts such as tape, probes, and batteries will be kept on site to reduce downtime. Backup instruments and equipment such as sensors will be available on site or within 1–day shipment to avoid delays in the field schedule. Table 15 presents a schedule for preventive maintenance for field equipment.

11.2 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

As part of its QA programs, routine preventive maintenance programs are conducted by Chemron Laboratories to minimize the occurrence of instrument failure and other system malfunctions. The laboratory has routine maintenance scheduled by vendors and coordinates with the vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications. Specific maintenance procedures are addressed in the laboratory QA Plans and SOPs.

Chemron's spare parts are inventoried and replaced by their Purchasing Agents. The system requires each analyst to formally check out replacement parts and log the inventory number, cost and vendor replacement parts and log the inventory number, cost and vendor information on log sheets. When the inventory of a specific item falls below a predetermined "critical" level, the Purchasing Agent automatically orders replacements in a time frame consistent with the laboratory's need. Overnight shipment capabilities are requested from all

vendors for maximum flexibility. As a result, laboratory personnel are not required to maintain a comprehensive parts list.

This system allows minimum spare parts to be stocked, which reduces costs while maintaining adequate surplus materials for routine needs. This system is also used for the purchase of solvents and many chemical standards.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

12.1 FIELD MEASUREMENTS

Field precision and accuracy criteria also are discussed in Sections 3.1.2 and 3.2.2, respectively.

Field data will be assessed by the Field Team Leader. Accuracy of the field measurements will be assessed using daily instrument calibration and calibration checks. Precision will be assessed on the basis of reproducibility of multiple readings for a single sample. The objective for data completeness for field measurements will be 90 percent.

12.2 LABORATORY MEASUREMENTS

Laboratory precision and accuracy criteria also are discussed in Sections 3.1.3 and 3.2.3, respectively.

12.2.1 Laboratory Accuracy Assessment

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analytes to be evaluated. An LCS also will be prepared for every 20 samples analyzed. A sample spike and LCS will be included in every set of 20 samples tested on each instrument. The spike sample and LCS are then analyzed. The increase in concentration of the analyte observed in the spiked sample or LCS due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample or clean matrix, determines the percent recovery. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{Amount\ in\ Spiked\ Sample - Amount\ in\ Sample}{Amount\ of\ the\ Spike\ Added} \times 100$$

12.2.2 Laboratory Precision Assessment

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{|Amount\ in\ Spike\ 1 - Amount\ in\ Spike\ 2|}{0.5\,(Amount\ in\ Spike\ 1 + Amount\ in\ Spike\ 2)} \times 100$$

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results (all those not coded "R") to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$Completeness = \left(\frac{number\ of\ valid\ measurements}{number\ of\ measurements\ planned}\right) \times 100$$

12.4 SENSITIVITY

The method detection limits (MDL) depend on instrumental sensitivity and matrix effects. The instrumental sensitivity will be determined through the analysis of method blank, calibration check sample, and laboratory control samples in accordance with SW–846 methodology. Analytes that employ EPA–600/4–79/020 (MCAWW) methods will comply with 40 CFR Part 136, Appendix B, Revision 1.1.1.

13.0 CORRECTIVE ACTION

This task describes the corrective action procedures to be used by Dames & Moore and the laboratory.

13.1 CORRECTIVE ACTION

Corrective actions may be required for two classes of problems: analytical and equipment problems, and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Dames & Moore DO Manager. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by a stop–work order by the DO Manager or Site Quality Assurance Manager.

13.2 FIELD CORRECTIVE ACTION

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Dames & Moore DO Manager or designee. The Dames & Moore DO Manager will be responsible for assessing the suspected problems, in consultation with the Site Quality Assurance Manager, based on the potential for the situation to impact the quality of the data. If it is determined that the situation will result in nonconformance with the

project plans and will require corrective action, a nonconformance report will be initiated by the DO Manager.

The DO Manager will be responsible for ensuring that corrective actions are initiated for nonconformances by:

1. Evaluating all reported nonconformances;
2. Controlling additional work on nonconforming items;
3. Determining disposition or action to be taken;
4. Maintaining a log of nonconformances;
5. Reviewing nonconformance reports and corrective actions taken; and
6. Ensuring nonconformance reports are included in the final facility documentation in project files.

If appropriate, the DO Manager will ensure that no additional work that is independent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

1. Repeat the measurement to check the error;
2. Check for all proper adjustments for ambient conditions such as temperature;
3. Check the batteries;
4. Check the calibration;
5. Replace the instrument or measurement devices;
6. Stop work.

The Project Coordinator or designee is responsible for all base activities. In this role, the Project Coordinator is required to adjust the base programs to accommodate base specific needs. When it becomes necessary to modify a program, the responsible person notifies the SC Project Coordinator of the anticipated change and implements the necessary changes after obtaining the approval of the Project Coordinator. The change in the program will be documented on the field change request (FCR) that will be signed by the initiators and the Project Manager. The FCR shall be attached to the file copy of the affected document. The Project Coordinator must approve the change in writing or verbally prior to field

implementation, if feasible. If unacceptable, the action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken.

The Dames & Moore Delivery Order Manager is responsible for controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties.

13.3 LABORATORY CORRECTIVE ACTION

Corrective actions are required whenever an out–of–control event or potential out–of–control event is noted. The investigative action taken is dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the Laboratory QAO during internal or external audits or from the results of performance evaluation samples;
- LCS recoveries are outside control limits; or
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst. All corrective actions will be documented and approved by the laboratory QAO or the QA designee.

The AFCEE has made some allowances to be exercised on a one–time basis if the problem is identified after a batch of samples have been analyzed. Specific guidance from the Handbook regarding correctiveactions in the laboratory are:

- The presence of analytes in a method blank at concentrations greater than the PQL approved in the SAP indicates a need for corrective action. Corrective actions shall be performed to eliminate the source of contamination prior to proceeding with analysis. No analytical data shall be corrected for the presence of analysis in blanks. When an analyte is detected in the blank, but not in the associated samples, no corrective action is necessary.

- Whenever an analyte in a LCS is outside of the recovery acceptance limit, data for the analyte may not be reported. All samples in the analytical batch must be reanalyzed for the out–of–control analyte after the system problems have been resolved and system control has been reestablished. When an analyte in a LCS exceeds the upper control limit and that analyte is not detected in the associated samples, the data for that analyte do not need to be qualified and no corrective action is required.

- Matrix spikes are used to evaluate the matrix effect, not to control the analytical process. The recoveries of analytes in the matrix spikes should be compared to the acceptance limits of those analytes in LCSs. If analytes in the matrix spike are outside the LCS control limits, a reanalysis of the matrix spike for those analytes is required. The matrix spike reanalysis is intended to evaluate whether the out–of–control event is specific to the handling of that particular sample. If the reanalysis is also out of control for the same analytes, matrix effect is confirmed and analytes in all related samples must be qualified. If the reanalysis is in control, the associated samples will not be qualified.

- When the recovery of a surrogate exceeds the acceptance limit, the corrective actions outlined in Section 8.10.5 of method SW8000 in SW–846 should be performed. Reextractions, if necessary, must be done within the holding times.

- All corrective actions to out–of–control events during laboratory analyses must be summarized and reported in the ITIRs and in the technical report (TR).

Corrective actions for laboratory problems are specified in the Chemron SOPs and QA manuals. Specific QC procedures are designed to help analysts determine the need for corrective action. Often, personal experience is most valuable in alerting the analyst to suspicious data or malfunctioning equipment. Corrective action taken at this point helps to avoid collection of poor quality data.

Problems not immediately detected during the course of analysis may require more formalized, long–term corrective action. The essential steps in the corrective action systems are as follows:

1. Identify and define the problem.
2. Assign responsibility for investigating the problem.
3. Investigate and determine the cause of the problem.
4. Determine a corrective action to eliminate the problem.
5. Assign and accept responsibility for implementing the corrective action.
6. Establish effectiveness of the corrective action and implement it.
7. Verify that the corrective action has eliminated the problem.

This scheme generally is accomplished through a request to the Site Quality Assurance Manager. Any laboratory analyst or project member may notify the Site Quality Assurance Manager of a problem. The Site Quality Assurance Manager initiates the corrective action scheme by relating the problem to the appropriate team managers and/or laboratory staff, who investigate or assign responsibility for investigating the problem and its cause. Once determined, an appropriate corrective action is approved by the Site Quality Assurance Manager. Its implementation is later verified through an audit. All corrective actions taken by a laboratory will be noted in the case narrative submitted to Dames & Moore with each data package.

13.4 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The Dames & Moore DO Manager/Site Quality Assurance Manager may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, and whether the data to be collected are necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded, etc.). When the Dames & Moore QAO identifies a corrective action situation, it is the AFCEE/ERB COR who will be responsible for approving the implementation of corrective actions with budget and schedule implications.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The DO specifies that monthly Progress Reports are required. The reports will include summaries of all problems or potential problems encountered during the reporting period and actions being taken to rectify problems. Accordingly, in order to provide comprehensive information to complete this report, periodic QA Reports summarizing the project quality achieved and corrective actions taken will be prepared by the DO Manager with input from the Dames & Moore Site Quality Assurance Manager and laboratory QAO.

In addition, the Draft and Final Reports will contain an assessment of the project compliance with the QAPP and FSP, and any limitations of the data obtained. Reconciliation of the data against the project DQOs and a summary of the periodic QA Reports will also be included.

14.1 CONTENTS OF PROJECT QA REPORTS

QA Reports will contain the results of field systems and laboratory on–site systems audits, and the results of laboratory performance evaluation samples analyzed within the time frame of the project samples. Corrective actions taken in response to these audits and their effectiveness will be included with an assessment of their effect on project data quality. The status of the project analytical work and data validation activities will be indicated. When appropriate, anticipated field or laboratory issues which may affect the project and proposed solutions will be addressed, and any QAPP modifications needed will be highlighted. Personnel changes in key positions and training that affects project operations will be included.

All QA Reports will be prepared by the Site Quality Assurance Manager and addressed to the Project Manager. If immediate corrective action to a deficiency found during auditing or data validation is needed, QA Reports will be made verbally by the QAO to the Project Manager, and will be documented in a Telephone Conversation Record format. All verbal QA Reports will be discussed in detail in the next QA Report.

14.2 FREQUENCY OF QA REPORTS

The QA Reports will be prepared monthly and submitted to the Project Manager one week prior to the due date of the Progress Report. The reports will commence upon approval of the Work Plans and end with the submittal of a Final Report. The frequency of interim verbal QA Reports cannot be estimated at this time. The Draft and Final Report QA sections will be prepared at the direction of the Project Manager to coincide with the preparation of the total report.

14.3 INDIVIDUALS RECEIVING/REVIEWING QA REPORTS

QA Reports will be reviewed by the laboratory QAO and Field Team Leader for accuracy of existing conditions and status of corrective actions. The DO Manager will be responsible for the distribution of the QA Report to project personnel as indicated in the organizational chart and for its incorporation into the monthly Progress Reports.

REFERENCES CITED

USEPA Contract Laboratory Program Statement of Work for Organic Analyses Multi–Media, Multi–Concentration, Document Numbers OLM01.0, OLM01.1, OLM01.1.1, February 1991.

Weast, Robert C., 1984, CRC Handbook of Chemistry and Physics, CRC Press Inc., Boca Raton, Florida. (Approximately 2,400 pp.)

AFCEE, Handbook for the Installation Restoration Program (IRP) Remedial Investigation/Feasibility Studies (RI/FS), September, 1993.

TABLE 1

Richards-Gebaur AFB, MO
PHASE I PRELIMINARY ASSESSMENT

TABLE 1

ASSESSMENT PARAMETERS AND RATIONALE
DEVELOPMENT OF QUALITY PROGRAM PLAN
RICHARDS-GEBAUR AFB, MO

SAMPLING LOCATION	ESTIMATED NUMBER OF LABORATORY SAMPLES AND MEDIA	ANALYTICAL PARAMETERS	RATIONALE	ESTIMATED NUMBER OF FIELD SAMPLES AND MEDIA	FIELD PARAMETERS	RATIONALE

TABLE 2

Richards-Gebaur AFB, MO
NUMBER/TYPE OF SAMPLES PROPOSED
TO ESTABLISH BACKGROUND

TABLE 2

NUMBER/TYPE OF SAMPLES
PROPOSED TO ESTABLISH BACKGROUND
DEVELOPMENT OF QUALITY PROGRAM PLAN
RICHARDS-GEBAUR AFB, MO

SAMPLE TYPE	NUMBER OF SAMPLES
Soil	n =
Sediment	n =
Surface Water	n =
Pore Water	n =
Ground Water	n =

TABLE 3

Richards-Gebaur AFB, MO
ANALYTICAL PARAMETERS AND REPORTING LIMITS

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Alkalinity A2320(W)	Carbonate	10	mg/L	-	mg/kg
	Bicarbonate	10	mg/L	-	mg/kg
	Hydroxide	10	mg/L	-	mg/kg
Radioactivity SW9310	Gross Alpha & Gross Beta	4	pCi/L(c)	(d)	pCi/g
SW9315	Radium 226	1	pCi/L(c)	(d)	pCi/g
SW9320	Radium 228	3	pCi/L(c)	(d)	pCi/g
Residue, Filterable E160.1(W)	Total Dissolved Solids	10	mg/L	-	mg/kg
Residue, Nonfilterable E160.2(W)	Total Suspended Solids	5	mg/L	-	mg/kg
Common Anions SW9058	Chloride	0.2	mg/L	-	mg/kg
	Floride	0.2	mg/L	-	mg/kg
	Sulfate	0.2	mg/L	-	mg/kg
	Nitrate	0.1	mg/L	-	mg/kg
	Ortho-Phosphate	0.1	mg/L	-	mg/kg
Nitrogen, Nitrate+Nitrite E353.1(W) E353.2(W)	Nitrate+Nitrite	0.1	mg/L	-	mg/kg
SW3020/SW7041(W) SW3050/SW7041(S)	Antimony	0.005	mg/L	0.5	mg/kg
SW7060(W&S)	Arsenic	0.005	mg/L	0.5	mg/kg
SW3020/SW7131(W) SW3050/SW7131(S)	Cadmium	0.001	mg/L	0.1	mg/kg
SW3020/SW7191(W) SW3050/SW7191(S)	Chromium	0.005	mg/L	0.5	mg/kg
SW3020/SW7421(W) SW3050/SW7421(S)	Lead	0.005	mg/L	0.5	mg/kg
SW7470(W) SW7471(S)	Mercury	0.001	mg/L	0.1	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
SW7740 (W&S)	Selenium	0.005	mg/L	0.5	mg/kg
SW3020/SW7841(W) SW3050/SW7841(S)	Thallium	0.001	mg/L	0.1	mg/kg
SW3020/SW7911(W) SW3050/SW7911(S)	Vanadium	0.004	mg/L	0.4	mg/kg
Inductively Coupled Plasma (ICP) Screen for Metals SW3005/SW6010(W) SW3050/SW6010(S)	Aluminum	0.5	mg/L	50	mg/kg
	Antimony	0.4	mg/L	40	mg/kg
	Arsenic	0.6	mg/L	60	mg/kg
	Barium	0.02	mg/L	2	mg/kg
	Beryllium	0.003	mg/L	0.3	mg/kg
	Cadmium	0.04	mg/L	4	mg/kg
	Calcium	0.1	mg/L	10	mg/kg
	Chromium	0.07	mg/L	7	mg/kg
	Cobalt	0.07	mg/L	7	mg/kg
	Copper	0.06	mg/L	6	mg/kg
	Iron	0.07	mg/L	7	mg/kg
	Lead	0.5	mg/L	50	mg/kg
	Magnesium	0.3	mg/L	30	mg/kg
	Manganese	0.02	mg/L	2	mg/kg
	Molybdenu	0.08	mg/L	8	mg/kg
	Nickel	0.15	mg/L	15	mg/kg
	Potassium	5	mg/L	500	mg/kg
	Selenium	0.8	mg/L	80	mg/kg
	Silver	0.07	mg/L	7	mg/kg
	Sodium	0.3	mg/L	30	mg/kg
	Thallium	0.4	mg/L	40	mg/kg
	Vanadium	0.08	mg/L	8	mg/kg
	Zinc	0.02	mg/L	2	mg/kg
SW9010(W) or SW9012(W)	Cyanide (total)	0.02	mg/L	-	mg/kg

a. If a lower action level or maximum contamination level for any given analyte is applicable to a specific project, the PQL may not exceed 50 percent of the regulating action level or maximum contamination level.
b. PQLs given are for the low-level requirements. Mid- and high-level requirements would have a corresponding adjustment in limits that will be specified in the SAP. These high levels shall be verified by the procedure specified for the low-level PQLs.
c. Report must include calibration standards and precision data.
d. Establish PQLs prior to analysis, or specify in the SAP.

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
1,2 Dibromoethane (EDB) See SW8260					
Petroleum Hydrocarbons E418.1(W)SW3550/ E418.1(S)(Mod)	(Not recommended for use due to requirement for Freon 113 extraction)	1	mg/L	30	mg/kg
SW5030/SW8015(Mod)	Gasoline	0.1	mg/L	1	mg/kg
SW3550/SW8015(Mod)	Diesel, Jet Fuel	1	mg/L	10	mg/kg
Purgeable Halocarbons SW5030/SW8010 (W&S)	Bromobenzene	5	µg/L	0.05	mg/kg
	Bromodichloromethane	1	µg/L	0.005	mg/kg
	Bromoform	2	µg/L	0.05	mg/kg
	Bromomethane	10	µg/L	0.01	mg/kg
	Carbon tetrachloride	1	µg/L	0.005	mg/kg
	Chlorobenzene	2.5	µg/L	0.005	mg/kg
	Chloroethane	5	µg/L	0.005	mg/kg
	Chloroform	0.5	µg/L	0.005	mg/kg
	1-Chlorohexane	5	µg/L	0.005	mg/kg
	2-Chloroethyl vinyl ether	10	µg/L	0.01	mg/kg
	Chloromethane	1	µg/L	0.005	mg/kg
	Dibromochloromethane	1	µg/L	0.005	mg/kg
	Dibromomethane	5	µg/L	0.005	mg/kg
	1,2-Dichlorobenzene	2	µg/L	0.005	mg/kg
	1,3-Dichlorobenzene	3	µg/L	0.005	mg/kg
	1,4-Dichlorobenzene	2	µg/L	0.005	mg/kg
	1,1-Dichloroethane	1	µg/L	0.005	mg/kg
	1,2-Dichloroethane	1	µg/L	0.005	mg/kg
	1,1-Dichloroethene	1	µg/L	0.005	mg/kg
	cis-1,2-Dichloroethene	1	µg/L	0.005	mg/kg
	trans-1,2-Dichloroethene	1	µg/L	0.005	mg/kg
	cis-1,3-Dichloropropene	5	µg/L	0.005	mg/kg
	1,2-Dichloropropane	1	µg/L	0.005	mg/kg
	trans-1,3-Dichloropropene	3	µg/L	0.005	mg/kg
	Methylene chloride	2	µg/L	0.005	mg/kg
	1,1,1,2-Tetrachloroethane	5	µg/L	0.005	mg/kg
	1,1,2,2-Tetrachloroethane	1	µg/L	0.005	mg/kg
	Tetrachloroethene	1	µg/L	0.005	mg/kg
	1,1,1-Trichloroethane	1	µg/L	0.005	mg/kg
	1,1,2-Trichloroethane	1	µg/L	0.005	mg/kg
	Trichloroethene	1	µg/L	0.005	mg/kg
	Trichlorofluoromethane	1	µg/L	0.005	mg/kg
	Trichloropropane	10	µg/L	0.01	mg/kg
	Vinyl Chloride	2	µg/L	0.005	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Nonhalogenated Volatile Organics SW5030/SW8015 (W&S)	Diethyl ether	50	µg/L	(c)	mg/kg
	Methyl ethyl ketone (MEK)	50	µg/L		mg/kg
	Methyl isobutyl ketone (MIBK)	50	µg/L		mg/kg
Purgeable Aromatic Volatiles SW5030/SW8020 (W&S)	Benzene	2	µg/L	0.002	mg/kg
	Chlorobenzene	2	µg/L	0.002	mg/kg
	1,2-Dichlorobenzene	4	µg/L	0.004	mg/kg
	1,3-Dichlorobenzene	4	µg/L	0.004	mg/kg
	1,4-Dichlorobenzene	3	µg/L	0.003	mg/kg
	Ethylbenzene	2	µg/L	0.002	mg/kg
	Toluene	2	µg/L	0.002	mg/kg
	Xylenes	2	µg/L	0.002	mg/kg
Organochlorine Pesticides & PCBs SW3510/SW8080(W) SW3550/SW8080(S)	Aldrin	0.04	µg/L	0.003	mg/kg
	alpha-BHC	0.03	µg/L	0.002	mg/kg
	beta-BHC	0.06	µg/L	0.004	mg/kg
	delta-BHC	0.09	µg/L	0.006	mg/kg
	Lindane (gamma-GHC)	0.04	µg/L	0.003	mg/kg
	Chlordane	0.14	µg/L	0.009	mg/kg
	4,4'-DDD	0.11	µg/L	0.007	mg/kg
	4,4'-DDE	0.04	µg/L	0.003	mg/kg
	4,4'-DDT	0.12	µg/L	0.008	mg/kg
	Dieldrin	0.02	µg/L	0.01	mg/kg
	Endosulfan I	0.14	µg/L	0.009	mg/kg
	Endosulfan II	0.04	µg/L	0.003	mg/kg
	Endosulfan sulfate	0.66	µg/L	0.04	mg/kg
	Endrin	0.06	µg/L	0.004	mg/kg
	Endrin aldehyde	0.23	µg/L	0.02	mg/kg
	Heptachlor	0.03	µg/L	0.002	mg/kg
	Heptachlor epoxide	0.83	µg/L	0.06	mg/kg
	Methoxychlor	1.76	µg/L	0.1	mg/kg
	Toxaphene	2.4	µg/L	0.2	mg/kg
	PCB-1016	1	µg/L	1	mg/kg
	PCB-1221	1	µg/L	1	mg/kg
	PCB-1232	1	µg/L	1	mg/kg
	PCB-1242	1	µg/L	1	mg/kg
	PCB-1248	1	µg/L	1	mg/kg
	PCB-1254	1	µg/L	1	mg/kg
	PCB-1260	1	µg/L	1	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Organophosphorus Pesticides SW3510/SW8140(W) SW3550/SW8140(S)	Azinphos methyl	15	µg/L	1	mg/kg
	Bolstar	1.5	µg/L	0.1	mg/kg
	Chorpyrifos	3	µg/L	0.2	mg/kg
	Coumaphos	15	µg/L	1	mg/kg
	Demeton-O	2.5	µg/L	0.2	mg/kg
	Demeton-S	2.5	µg/L	0.2	mg/kg
	Diazinon	6	µg/L	0.4	mg/kg
	Dichlorovos	10	µg/L	0.7	mg/kg
	Disulfoton	2	µg/L	0.1	mg/kg
	Ethoprop	2.5	µg/L	0.2	mg/kg
	Fensulfothion	15	µg/L	1	mg/kg
	Fenthion	1	µg/L	0.1	mg/kg
	Merphos	2.5	µg/L	0.2	mg/kg
	Mevinphos	3	µg/L	0.2	mg/kg
	Naled	1	µg/L	0.1	mg/kg
	Parathion methyl	0.3	µg/L	0.02	mg/kg
	Phorate	1.5	µg/L	0.1	mg/kg
	Ronnel	3	µg/L	0.2	mg/kg
	Stirophos	50	µg/L	2.4	mg/kg
	Tokuthion	5	µg/L	0.4	mg/kg
	Trichloronate	1.5	µg/L	0.1	mg/kg
Chlorinated Phenoxy Acid Herbicides SW8150 (W&S)	2,4-D	12	µg/L	0.8	mg/kg
	2,4-DB	9	µg/L	0.6	mg/kg
	2,4,5-T	2	µg/L	0.1	mg/kg
	2,4,5-TP	1.7	µg/L	0.1	mg/kg
	Dalapon	60	µg/L	4	mg/kg
	Dicamba	2.7	µg/L	0.2	mg/kg
	Dichloroprop	6.5	µg/L	0.5	mg/kg
	Dinoseb	0.7	µg/L	0.05	mg/kg
	MCPA	2500	µg/L	170	mg/kg
	MCPP	1900	µg/L	130	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Semivolatile Organic Compounds SW3510/SW8270(W) SW3550/SW8270(S)	Base/Neutral Extractibles				
	Acenapthene	10	µg/L	0.7	mg/kg
	Acenaphthylene	10	µg/L	0.7	mg/kg
	Anthracene	10	µg/L	0.7	mg/kg
	Benzo (a) anthracene	10	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10	µg/L	0.7	mg/kg
	Benzo (a) pyrene	10	µg/L	0.7	mg/kg
	Benzyl alcohol	20	µg/L	1.3	mg/kg
	bis (2-Chloroethoxy) methane	10	µg/L	0.7	mg/kg
	bis (2-Chlorethyl) ether	10	µg/L	0.7	mg/kg
	bis (2-Chloroiso-propyl) ether	10	µg/L	0.7	mg/kg
	bis (2-ethylhexyl) phthalate	10	µg/L	0.7	mg/kg
	4-Bromophenyl phenyl ether	10	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10	µg/L	0.7	mg/kg
	4-Chloroaniline	20	µg/L	1.3	mg/kg
	2-Chloronaphthalene	10	µg/L	0.7	mg/kg
	4-Chlorophenyl phenyl ether	10	µg/L	0.7	mg/kg
	Chrysene	10	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10	µg/L	0.7	mg/kg
	Dibenzofuran	10	µg/L	0.7	mg/kg
	Di-n-Butylphthalate	10	µg/L	0.7	mg/kg
	1,2-Dichlorobenzene	10	µg/L	0.7	mg/kg
	1,3-Dichlorobenzene	10	µg/L	0.7	mg/kg
	1,4-Dichlorobenzene	10	µg/L	0.7	mg/kg
	3.3'-Dichlorobenzidine	20	µg/L	1.3	mg/kg
	Diethyl phthalate	10	µg/L	0.7	mg/kg
	Dimethly phthalate	10	µg/L	0.7	mg/kg
	2,4-Dinitrotoluene	10	µg/L	0.7	mg/kg
	2,6-Dinitrotoluene	10	µg/L	0.7	mg/kg
	Di-n-octyl phthalate	10	µg/L	0.7	mg/kg
	Fluoranthene	10	µg/L	0.7	mg/kg
	Fluorene	10	µg/L	0.7	mg/kg
	Hexachlorobenzene	10	µg/L	0.7	mg/kg
	Hexachlorobutadiene	10	µg/L	0.7	mg/kg
	Hexachlorocyclopentadiene	10	µg/L	0.7	mg/kg
	Hexachloroethane	10	µg/L	0.7	mg/kg
	Indeno (q,w,e,-cd)pyrene	10	µg/L	0.7	mg/kg
	Isophorone	10	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10	µg/L	0.7	mg/kg
	Naphthalene	10	µg/L	0.7	mg/kg
	2-Nitroaniline	50	µg/L	3.3	mg/kg
	3-Nitroaniline	50	µg/L	3.3	mg/kg
	4-Nitroaniline	50	µg/L	3.3	mg/kg
	Nitrobenzene	10	µg/L	0.7	mg/kg
	n-Nitrosodiphenyl-amine	10	µg/L	0.7	mg/kg
	n-Nitrosodipropyl-amine	10	µg/L	0.7	mg/kg
	Phenanthrene	10	µg/L	0.7	mg/kg
	Pyrene	10	µg/L	0.7	mg/kg
	1.2.4-Trichlorobenzene	10	µg/L	0.7	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Semivolatile Organic Compounds (Concluded) SW3510/SW8270(W) SW3550/SW8270(S)	Acid Extractables				
	Benzoic acid	50	µg/L	1.6	mg/kg
	4-Chloro-3-methylphenol	20	µg/L	1.3	mg/kg
	2-Chlorophenol	10	µg/L	0.3	mg/kg
	2,4-Dichlorophenol	10	µg/L	0.3	mg/kg
	2,4-Dimethylphenol	10	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50	µg/L	3.3	mg/kg
	2,4-Dinitrophenol	50	µg/L	3.3	mg/kg
	2-Methylphenol	10	µg/L	0.3	mg/kg
	4-Methylphenol	10	µg/L	0.3	mg/kg
	2-Nitrophenol	10	µg/L	0.3	mg/kg
	4-Nitrophenol	50	µg/L	1.6	mg/kg
	Pentachlorophenol	50	µg/L	3.3	mg/kg
	Phenol	10	µg/L	0.3	mg/kg
	2,4,5-Trichlorophenol	50	µg/L	3.3	mg/kg
	2,4,6-Trichlorophenol	10	µg/L	0.3	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Units	Soil[b] PQL	Soil[b] Units
Volatile Organic Compounds SW8260(d)	Dichlorodifluoromethane	.6	µg/L	.004	mg/kg
	Chloromethane	1.0	µg/L	.006	mg/kg
	Vinyl chloride	.6	µg/L	.004	mg/kg
	Bromomethane	1.2	µg/L	.008	mg/kg
	Chloroethane	1.0	µg/L	.006	mg/kg
	Trichlorofluoromethane	1.0	µg/L	.006	mg/kg
	1,1-Dichloroethene	1.0	µg/L	.006	mg/kg
	Methylene chloride	.8	µg/L	.005	mg/kg
	trans-1,2-Dichloroethene	.8	µg/L	.005	mg/kg
	1,1-Dichloroethane	.6	µg/L	.004	mg/kg
	2,2-Dichloropropane	.6	µg/L	.004	mg/kg
	cis-1,2-Dichloroethene	.8	µg/L	.005	mg/kg
	Chloroform	.6	µg/L	.004	mg/kg
	Bromochloromethane	1.2	µg/L	.008	mg/kg
	1,1,1-Trichloroethane	1.0	µg/L	.006	mg/kg
	Carbon tetrachloride	.6	µg/L	.004	mg/kg
	1,1-Dichloropropene	1.0	µg/L	.006	mg/kg
	Benzene	.6	µg/L	.004	mg/kg
	1,2-Dichloroethane	.6	µg/L	.004	mg/kg
	Trichloroethene	.4	µg/L	.003	mg/kg
	1,2-dichloropropane	.4	µg/L	.003	mg/kg
	Bromodichloromethane	.6	µg/L	.004	mg/kg
	Dibromomethane	.4	µg/L	.003	mg/kg
	trans-1,3-Dichloropropene	.6	µg/L	.004	mg/kg
	Toluene	.6	µg/L	.004	mg/kg
	cis-1,3-Dichloropropene	.4	µg/L	.003	mg/kg
	1,1,2-Trichloroethane	.6	µg/L	.004	mg/kg
	Tetrachloroethene	1.0	µg/L	.006	mg/kg
	1,3-Dichloropropane	.6	µg/L	.004	mg/kg
	Dibromochloromethane	.6	µg/L	.004	mg/kg
	1,2-Dibromoethane (EDB)	.6	µg/L	.004	mg/kg
	1-Chlorohexane	1.0	µg/L	.006	mg/kg
	Chlorobenzene	.8	µg/L	.005	mg/kg
	1,1,1,2-Tetrachloroethane	1.0	µg/L	.006	mg/kg
	Ethylbenzene	1.0	µg/L	.006	mg/kg
	p-Xylene	1.0	µg/L	.006	mg/kg
	m-Xylene	1.0	µg/L	.006	mg/kg
	o-Xylene	.8	µg/L	.005	mg/kg
	Styrene	.8	µg/L	.005	mg/kg
	Bromoform	.4	µg/L	.003	mg/kg
	Isopropylbenzene	.5	µg/L	.006	mg/kg
	1,1,2,2-Tetrachloroethane	.6	µg/L	.004	mg/kg
	Bromobenzene	1.0	µg/L	.006	mg/kg
	1,2,3-Trichloropropane	.6	µg/L	.004	mg/kg
	n-Propylbenzene	.5	µg/L	.006	mg/kg
	2-Chlorotoluene	1.2	µg/L	.008	mg/kg
	1,3,5-Trimethylbenzene	.4	µg/L	.003	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Units	Soil[b] PQL	Soil[b] Units
Volatile Organic Compounds (Concluded) SW8260(d)	4-Chlorotoluene	1.2	µg/L	.008	mg/kg
	tert-Butylbenzene	2.0	µg/L	.014	mg/kg
	1,2,4-Trimethylbenzene	.4	µg/L	.003	mg/kg
	sec-Butylbenzene	2.0	µg/L	.014	mg/kg
	p-Isopropyltoluene	.5	µg/L	.006	mg/kg
	1,3-Dichlorobenzene	1.0	µg/L	.006	mg/kg
	1,4-Dichlorobenzene	1.2	µg/L	.008	mg/kg
	n-Butylbenzene	2.0	µg/L	.014	mg/kg
	1,2-Dichlorobenzene	1.0	µg/L	.006	mg/kg
	1,2-Dibromo-3-chloropropane (DBCP)	1.2	µg/L	.008	mg/kg
	1,2,4-Trichlorobenzene	1.2	µg/L	.008	mg/kg
	Hexachlorobutadiene	5.0	µg/L	.03	mg/kg
	Naphthalene	5.0	µg/L	.030	mg/kg
	1,2,3-Trichlorobenzene	1.2	µg/L	.008	mg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Volatile Organic Compounds SW8240 (W&S)	Acetone	100	µg/L	0.1	mg/kg
	Benzene	5	µg/L	0.005	mg/kg
	Bromodichloromethane	5	µg/L	0.005	mg/kg
	Bromoform	5	µg/L	0.005	mg/kg
	Bromomethane	10	µg/L	0.01	mg/kg
	2-Butanone (MEK)	100	µg/L	0.1	mg/kg
	Carbon disulfide	5	µg/L	0.005	mg/kg
	Carbon tetrachloride	5	µg/L	0.005	mg/kg
	Chlorobenzene	5	µg/L	0.005	mg/kg
	Dibromochloromethane	5	µg/L	0.005	mg/kg
	Chloroethane	10	µg/L	0.01	mg/kg
	2-Chloroethyl vinyl ether	10	µg/L	0.01	mg/kg
	Chloroform	5	µg/L	0.005	mg/kg
	Chloromethane	10	µg/L	0.01	mg/kg
	1,1-Dichloroethane	5	µg/L	0.005	mg/kg
	1,2-Dichloroethane	5	µg/L	0.005	mg/kg
	1,1-Dichloroethene	5	µg/L	0.005	mg/kg
	cis-1,2-Dichloroethene	5	µg/L	0.005	mg/kg
	trans-1,2-Dichloroethene	5	µg/L	0.005	mg/kg
	1,2-Dichloropropane	5	µg/L	0.005	mg/kg
	cis-1,3-Dichloropropene	5	µg/L	0.005	mg/kg
	trans-1,3-Dichloropropene	5	µg/L	0.005	mg/kg
	Ethylbenzene	5	µg/L	0.005	mg/kg
	2-Hexanone	50	µg/L	0.05	mg/kg
	Methylene chloride	5	µg/L	0.005	mg/kg
	4-Methyl-2-pentanone (MIBK)	50	µg/L	0.05	mg/kg
	Styrene	5	µg/L	0.005	mg/kg
	1,1,2,2-Tetrachloroethane	5	µg/L	0.005	mg/kg
	Tetrachloroethene	5	µg/L	0.005	mg/kg
	Toluene	5	µg/L	0.005	mg/kg
	1,1,1-Trichloroethane	5	µg/L	0.005	mg/kg
	1,1,2-Trichloroethane	5	µg/L	0.005	mg/kg
	Trichloroethene	5	µg/L	0.005	mg/kg
	Vinyl acetate	50	µg/L	0.05	mg/kg
	Vinyl chloride	10	µg/L	0.01	mg/kg
	Xylenes (total all isomers)	5	µg/L	0.005	mg/kg
Dioxins & Furans SW8280 (W &S)	2,3,7,8-TCDD	0.44	ng/L	0.17	µg/kg
SW8290 (W &S)	2,3,7,8-TCDD	0.01	ng/L	0.001	µg/kg

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter/Method	Analyte	Water[b] PQL	Water[b] Unit	Soil[b] PQL	Soil[b] Unit
Explosives SW8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	13	µg/L	2.2	mg/kg
	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	14	µg/L	1	mg/kg
	1,3,5-Trinitrobenzene (TNB)	7.3	µg/L	0.25	mg/kg
	1,3-Dinitrobenzene (DNB)	4	µg/L	0.25	mg/kg
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	44	µg/L	0.65	mg/kg
	Nitrobenzene	7	µg/L	0.26	mg/kg
	2,4,6-Trinitrotoluene	6.9	µg/L	0.25	mg/kg
	2,4-Dinitrotoluene	5.7	µg/L	0.25	mg/kg
	2,6-Dintrotoluene	9.4	µg/L	0.26	mg/kg
	o-Nitrotoluene	12	µg/L	0.25	mg/kg
	m-Nitrotoluene	7.9	µg/L	0.25	mg/kg
	p-Nitrotoluene	8.5	µg/L	0.25	mg/kg
Polynuclear Aromatic Hydrocarbons SW3510/ SW8310(W) SW3550/ SW8310(S)	Naphthalene	18	µg/L	1.2	mg/kg
	Acenaphthylene	23	µg/L	1.54	mg/kg
	Acenaphthene	18	µg/L	1.2	mg/kg
	Fluorene	2.1	µg/L	0.14	mg/kg
	Phenanthrene	6.4	µg/L	0.42	mg/kg
	Anthracene	6.6	µg/L	0.44	mg/kg
	Fluoranthrene	2.1	µg/L	0.14	mg/kg
	Pyrene	2.7	µg/L	0.18	mg/kg
	Benzo(a)anthracene	0.13	µg/L	0.009	mg/kg
	Chrysene	1.5	µg/L	0.1	mg/kg
	Benzo(b)fluoranthene	0.18	µg/L	0.012	mg/kg
	Benzo(k)fluoranthene	0.17	µg/L	0.011	mg/kg
	Benzo(a)pyrene	0.23	µg/L	0.015	mg/kg
	Dibenzo(a,h)anthra-cene	0.3	µg/L	0.02	mg/kg
	Benzo(g,h,i)perylene	0.76	µg/L	0.05	mg/kg
	Indeno(1.2.3-cd) pyrene	0.43	µg/L	0.03	mg/kg
SW9065(W)	Phenols (total)	0.02	mg/L	-	

a. If a lower action level or maximum contamination level for any given analyte is applicable to a specific project, the PQL may not exceed 50 percent of the regulating action level or maximum contamination level.
b. PQLs given are for the low-level requirements. Mid- and high-level requirements would have a corresponding adjustment in limits that will be specified in the SAP. These specified high levels shall be verified by the procedure specified for the low-level PQLs.
c. Establish PQLs prior to analysis, or specify in the SAP.
d. This method may be substituted for method SW8240 on a project-specific basis when lower reporting limits are required.

TABLE 3

PRACTICAL QUANTITATION LIMITS INORGANIC ANALYTES (Continued)

Parameter	Method	Analyte	PQL/Units
Volatile Organic Compounds, non-polar	TO-1, TO-2	(a)	(b)
Organochlorine Pesticides & PCBs	TO-4	(a)	(b)
Phenols	TO-8	(a)	(b)
Dioxins	TO-9	(a)	(b)
Polynuclear Aromatic Hydrocarbons	TO-13	(a)	(b)
Volatile Organics in Connisters	TO-14	(a)	(b)

a. Listed in the "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air," June 1988, EPA/600/4-89/017.
b. Establish PQLs and units prior to analysis, or specify in the SAP.

TABLE 4

Richards-Gebaur AFB, MO
SAMPLING AND ANALYSIS SUMMARY TABLE

TABLE 4

SAMPLING AND ANALYSIS SUMMARY TABLE
DEVELOPMENT OF QUALITY PROGRAM PLAN
RICHARDS-GEBAUR AFB, MO

SAMPLE MATRIX	FIELD PARAMETERS	DQO LEVEL	LABORATORY PARAMETERS	DQO LEVEL	LABORATORY	FIELD						LABORATORY QC		
						FIELD SAMPLES	TB	FB	FD	MS/MSD	TOTAL TO LAB	MB	LCS	SCS
GROUND WATER SAMPLES														
SURFACE WATER SAMPLES														
SOIL														
SEDIMENT SAMPLES														

Notes:
* = included with ground water samples from new wells
** = included with water samples from sediment and river water
TB = Trip Blank
FB = Field Blank
MS/MSD = Matrix Spike/Matrix Spike Duplicate
MB = Method Blank
LCS = Laboratory Control Sample
SCS = Spiked Control Sample

Trip blanks are estimated. One trip blank will accompany each sample cooler containing samples to be analyzed for VOCs.

TABLE 5

Richards-Gebaur AFB, MO
ANALYTICAL RESPONSIBILITIES

TABLE 5

ANALYTICAL RESPONSIBILITIES
DEVELOPMENT OF QUALITY PROGRAM PLAN
RICHARDS-GEBAUR AFB, MO

ANALYTES	SOIL			SEDIMENT		GROUND WATER			SURFACE WATER	
	BACKGROUND LOCATIONS	WELL INSTALLATION BORINGS	SOIL BORINGS	RIVER	POND	BACKGROUND LOCATIONS	NEW WELLS/ PIEZOMETERS	EXISTING WELLS	STREAM	POND
ORGANICS										
METALS										
GEOTECHNICAL/PHYSICAL										
GEOCHEMICAL/WATER QUALITY										
FIELD PARAMETERS										

LABORATORIES:

D&M = Dames & Moore Soils Lab
CHEMRON = CHEMRON Environmental Laboratories
FIELD = In-field measurements

TABLE 6

Richards-Gebaur AFB, MO
PRECISION OBJECTIVES FOR FIELD DUPLICATES AND FIELD MEASUREMENTS

TABLE 6

PRECISION OBJECTIVES FOR FIELD DUPLICATES AND FIELD MEASUREMENTS
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

ANALYTE	FREQUENCY	DQO LEVEL	AQUEOUS LIMIT	SOIL/SEDIMENT LIMIT
Field Measurements				
Temperature	1/10	2	±0.5°C	±0.5°C
pH	1/10	2	20% or ±0.1 unit	20% or ±0.1 unit
Turbidity	1/10	2	20% or ±0.02 NTU	--
Conductivity	1/10	2	10% RPD	--
Geotechnical/Physical				
Grain size	1/10	3	--	
Density	1/10	3	--	
pH	1/10	3	--	20% or ±0.1 unit
Effective porosity	1/10	3	--	25% RPD or ±0.1 unit
Chemical				
Metals	1/10	0	35% RPD	50% RPD
Volatiles by 8260	1/20	4	133% RPD	133% RPD
Semivolatiles by 8270	1/20	0	133% RPD	133% RPD
PCB/pesticides by 8080	1/20	0	133% RPD	133% RPD
TDS	1/10	0	30% RPD	--
TSS	1/10	0	30% RPD	--
TOC	1/10	0	30% RPD	40% RPD
TPH	1/20	4	133% RPD	60% RPD
CEC	1/10	0	--	25% RPD

Notes: -- = Not applicable, not project analyte.

Frequency is expressed as number of field duplicates or duplicate measurements made per number of samples collected or measured.

$$RPD = \frac{(x-y)}{0.5(x+y)} \times 100$$

where:

RPD = Relative Percent Difference
x = the first analytical result
y = the second analytical result

TABLE 7

Richards-Gebaur AFB, MO
PRECISION OBJECTIVES FOR LABORATORY MS/MSD

TABLE 7
PRECISION AND ACCURANCE OBJECTIVES FOR ORGANIC ANALYTE LABORATORY MEASUREMENTS
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION %RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: WATER					
VOLATILE ORGANICS					
Benzene	8260	21	21	86-116	85-116
Chlorobenzene	8260	21	21	77-118	77-118
1,1-Dichloroethene	8260	22	22	75-130	75-130
Trichloroethene	8260	24	24	79-114	79-114
Toluene	8260	21	21	84-116	84-116
SEMIVOLATILE ORGANICS					
1,2,4-Trichlorobenzene	8270	28	28	25-109	25-109
Acenaphthene	8270	31	31	25-111	25-111
2,4-Dinitrotoluene	8270	38	38	33-109	33-109
Pyrene	8270	31	31	24-128	24-128
N-nitroso-di-n-propylamine	8270	38	38	12-112	18-123
1,4-Dichlorobenzene	8270	28	28	12-112	12-112
Pentachlorophenol	8270	50	50	18-113	18-113
Phenol	8270	42	42	2-87	2-87
2-Chlorophenol	8270	40	40	6-111	6-111
4-Chloro-3-methylphenol	8270	42	42	16-119	16-119
4-Nitrophenol	8270	31	31	11-119	11-119

Notes: MS/MSD = Matrix spike/matrix spike duplicate.
LCS = Laboratory control standard.

TABLE 7 (continued)

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION % RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: WATER					
HERBICIDES					
2,4-D	8150	50	50	35-150	35-150
2,4,5-TP (Silvex)	8150	50	50	8-148	8-148
2,4,5-T	8150	50	50	47-129	47-129

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION % RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: SOILS/SEDIMENTS					
HERBICIDES					
2,4-D	8150	50	50	35-150	35-150
2,4,5-TP (Silvex)	8150	50	50	8-148	8-148
2,4,5-T	8150	50	50	42-129	42-129

TABLE 7 (continued)

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION % RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: WATER					
PESTICIDES					
Gamma-BHC (Lindane)	8080	23	23	32-127	32-127
Heptachlor	8080	20	20	34-111	34-111
Aldrin	8080	21	21	42-122	42-122
Dieldrin	8080	38	38	36-146	36-146
Endrin	8080	37	37	30-147	30-147
4,4'-DDT	8080	36	36	25-160	25-160

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION % RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: SOILS/SEDIMENTS					
PESTICIDES					
Gamma-BHC (Lindane)	8080	29	29	D-116	D-116
Heptachlor	8080	28	28	D-136	D-136
Aldrin	8080	25	25	D-115	D-115
Dieldrin	8080	42	42	D-167	D-167
Endrin	8080	42	42	D-174	D-174
4,4'-DDT	8080	39	39	D-171	D171

TABLE 7 (continued)

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	LCS PRECISION % RSD	ACCURACY (% RECOVERY)	
				LCS	MS/MSD
MATRIX: SOILS/SEDIMENTS					
VOLATILE ORGANICS (LOW LEVEL)					
Benzene	8260	21	21	58-144	58-144
Chlorobenzene	8260	21	21	68-124	68-124
1,1-Dichloroethene	8260	22	22	29-149	29-149
Trichloroethene	8260	24	24	65-121	65-121
Toluene	8260	21	21	73-121	73-121
SEMIVOLATILE ORGANICS					
1,2,4-Trichlorobenzene	8270	23	23	15-104	15-104
Acenaphthene	8270	19	19	11-115	11-115
2,4-Dinitrotoluene	8270	47	47	26-100	26-100
Pyrene	8270	36	36	D-143	D-143
N-nitroso-di-n-propylamine	8270	38	38	17-108	17-108
1,4-Dichlorobenzene	8270	27	27	20-124	20-124
Pentachlorophenol	8270	47	47	D-122	D-122
Phenol	8270	35	35	3-109	3-109
2-Chlorophenol	8270	50	50	11-98	11-98
4-Chloro-3-methylphenol	8270	41	41	8-109	8-109
4-Nitrophenol	8270	50	50	D-158	D-158

TABLE 8

Richards-Gebaur AFB, MO
SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES

TABLE 8

SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES

Name	Methods of Analysis	Container[1]	Preservation[2,3]	Minimum Sample Volume or Weight	Maximum Holding Time
Inorganic tests					
Alkalinity (Field Test)	A2320	P,G	None Required	50 mL	Analyze immediately
Alkalinity (Lab Test)	A2320	P,G	4°C	50 mL	14 days
Common Anions	SW9056	P,G	None Required	50 mL	28 days for Br,F,CL,SO_4; 48 hrs for NO_3,NO_2,PO_4
Cyanide, Total, and Amenable to Chlorination	SW9010	P,G,T	4°C,NaOH to pH<12[2] 0.6 g ascorbic acid	500 mL or 4 ounces	14 days (water and soil)
Filterable Residue	E160.1	P,G	4°C	100 mL	7 days
Non-Filterable Residue	E160.2	P,G	4°C	100 mL	7 days
Hydrogen Ion (pH) w/s (Field Test)	SW9040/ SW9045	P,G	None Required	N/A	Analyze immediately
Nitrogen, Nitrate + Nitrite	E353.1	P,G	4°C, H_2SO_4 to pH<2	500 mL	28 days
Specific Conductance (Field Test)	SW9050	P,G	None Required	N/A	Analyze immediately
Temperature	E170.1	P,G	None Required	N/A	Analyze immediately
Total Organic Carbon	SW9060	P,G,T	4°C, HCl or H_2SO_4 to pH<2[2]	500 mL or 4 ounces	28 days (water and soil)
Metals					
Chromium VI	SW7196	P,G,T	4°C	500 mL or 8 ounces	24 hours (water and soil)[4]
Mercury	SW7470, SW7471	P,G,T	HNO_3 to pH<2[2], 4°C	500 mL or 8 ounces	28 days (water and soil)
Metals, except Chromium VI and Mercury	SW6010 and SW-846 atomic absorption methods	P,G,T	HNO_3 to pH<2[2], 4°C	500 mL or 8 ounces	180 days (water and soil)
Organic Tests					
Petroleum Hydrocarbons	E418.1[5]	G,T	4°C,,H_2SO_4 to pH<2[2]	1 Liter or 8 ounces	Water and soil--28 days
Fuel Hydrocarbons					
Volatile	SW8015 (modified)	G, Teflon-lined Septum T	4°C,,HCl to pH<2[2]	2x40 mL or 4 ounces	14 days (water and soil) 7 days if unpreserved by acid

TABLE 8

SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES (Continued)

Extractable	SW8015 (modified)	G,amber,T	4°C	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Aromatic Volatile Organics	SW8020	G,Teflon-lined, Septum, T	4°C, HCl to pH<2, 0.008% $Na_2S_2O_3$	2x40 mL or 4 ounces	14 days (water and soil), 7 days unpreserved by acid
Chlorinated Herbicides	SW8150	G, Teflon-lined, Cap, I	4°C, pH 5-9	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Pesticides and Polychlorinated Biphenyls (PCBs)	SW8080, SW8140	G, Teflon-lined, Cap T	4°C, pH 5-9	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Phenols	SW8040	G, Teflon-lined, Cap T	4°C, 0.008% $Na_2S_2O_3$	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Semivolatile Organics	SW8270	G, Teflon-lined Cap, T	4°C, 0.008% $Na_2S_2O_3$	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Volatile Organics	SW8240, SW8015(mod), SW8010 SW8260	G, Teflon-lined Septum, T	4°C, 0.008% $Na_2S_2O_3$ (HCl to pH<2 for volatile aromatics by SW8240)[2] and SW8260	2 x 40 mL or 4 ounces	14 days (water and soil); 7 days unpreserved by acid
Polycyclic Aromatic Hydrocarbons (PAHs)	SW8310	G, Teflon-lined Cap, T	4°C, store in dark, 0.008% $Na_2S_2O_3$	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Carbamate Pesticides	SW8314	G, Teflon-lined Cap, T	4°C, 0.008% $Na_2S_2O_3$	1 liter or 8 ounces	Water-7 days until extraction; 40 days after extraction Soil-14 days until extraction; 40 days after extraction
Dioxins	SW8280 SW8290	G, Teflon-lined Cap, T	4°C, 0.008% $Na_2S_2O_3$	1 liter or 8 ounces	Water and soils-30 days until extraction; 45 days after extraction
1,2-dibromoethane	E504	G, Teflon-lined Cap, T	4°C, 0.008% $Na_2S_2O_3$	2 x 40 mL	Water-28 days

TABLE 8

SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES (Continued)

Radiological Tests Alpha, Beta, and Radium	SW9310, SW9315, SW9320	P, G, T	HNO_3 to pH<2[2]	2 liters or 16 ounces	180 days
Toxicity Characteristic Leaching Procedure (TCLP)	SW1311	G, teflon-lined cap, T	Cool, 4°C	1 liter or 8 ounces	Volatiles-14 days to TCLP extraction; 14 days after extraction Semivolatiles-14 days to TCLP extraction; 40 days after prep. extraction Mercury-28 days to TCLP extraction; 28 days after extraction Metals-180 days to TCLP extraction; 180 days after extraction
Explosive Residues	SW-8330	P, G, T	Cool, 4°C	1 liter or 8 ounces	Water-7 days to extraction Soils-14 days to extraction Analysis-within 40 days after extraction

1. Polythylene (P); Glass (G); Brass sleeves in the sample barrel, sometimes called California Brass (T).
2. No pH adjustment for soil.
3. Preservation with 0.008% $Na_2S_2O_3$ is only required when residual chlorine is present.
4. Holding time for chromium VI in soils has not been established. The recommended holding time for extracting into water is 48 hours. The sample must be analyzed within 24 hours of extraction.
5. The use of E418.1 requires specific AFCEE approval. See Section 2.2.

TABLE 9

Richards-Gebaur AFB, MO
ACCURACY OBJECTIVES FOR FIELD MEASUREMENTS

TABLE 9

ACCURACY OBJECTIVES FOR FIELD MEASUREMENTS
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

FIELD ANALYTE	FREQUENCY	DQO LEVEL	TYPE OF ACCURACY QC CHECK	LIMIT
Temperature	annually	2	check thermometer against NIST reference	±0.5°C
pH	daily	2	LCS (buffers)	±0.1 s.u.
Turbidity	daily	2	LCS (40 NTU standards)	±4 NTU
Conductivity	daily	2	LCS (1413 KCl μmhos/cm)	10% of scale*
Total VOCs by PID	daily	1	calibration gas	±0.2 ppm

LCS = Laboratory control standard.

TABLE 10

Richards-Gebaur AFB, MO
ORGANIC ANALYTE LABORATORY MS/MSD
PRECISION AND ACCURACY OBJECTIVES

TABLE 10
PRECISION AND ACCURANCE OBJECTIVES FOR ORGANIC ANALYTE LABORATORY MEASUREMENTS
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	ACCURACY (% RECOVERY)	
			LCS	MS/MSD
MATRIX: WATER				
VOLATILE ORGANICS				
Benzene	8260	11	76-127	76-127
Chlorobenzene	8260	13	75-130	75-130
1,1-Dichloroethene	8260	14	61-145	61-145
Trichloroethene	8260	14	71-120	71-120
Toluene	8260	13	76-125	76-125
SEMIVOLATILE ORGANICS				
1,2,4-Trichlorobenzene	8270	28	25-109	25-109
Acenaphthene	8270	31	25-111	25-111
2,4-Dinitrotoluene	8270	38	33-109	33-109
Pyrene	8270	31	24-128	24-128
N-nitroso-di-n-propylamine	8270	38	18-123	18-123
1,4-Dichlorobenzene	8270	28	12-112	12-112
Pentachlorophenol	8270	50	18-113	18-113
Phenol	8270	42	2-87	2-87
2-Chlorophenol	8270	40	6-111	6-111
4-Chloro-3-methylphenol	8270	42	16-110	16-110
4-Nitrophenol	8270	31	D-119	D-119

Notes: MS/MSD = Matrix spike/matrix spike duplicate.
LCS = Laboratory control standard.

TABLE 10 (continued)

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	ACCURACY (% RECOVERY)	
			LCS	MS/MSD
MATRIX: SOILS/SEDIMENTS				
VOLATILE ORGANICS (LOW LEVEL)				
Benzene	8260	21	66-142	66-142
Chlorobenzene	8260	21	60-133	60-133
1,1-Dichloroethene	8260	22	59-172	59-172
Trichloroethene	8260	24	62-137	62-137
Toluene	8260	21	59-139	59-139
SEMIVOLATILE ORGANICS				
1,2,4-Trichlorobenzene	8270	23	15-104	15-104
Acenaphthene	8270	19	11-115	11-115
2,4-Dinitrotoluene	8270	47	26-100	26-100
Pyrene	8270	36	D-143	D-143
N-nitroso-di-n-propylamine	8270	38	17-108	17-108
1,4-Dichlorobenzene	8270	27	20-124	20-124
Pentachlorophenol	8270	47	D-122	D-122
Phenol	8270	35	3-109	3-109
2-Chlorophenol	8270	50	11-98	11-98
4-Chloro-3-methylphenol	8270	41	8-109	8-109
4-Nitrophenol	8270	50	D-158	D-158

TABLE 10 (continued)

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	ACCURACY (% RECOVERY)	
			LCS	MS/MSD
MATRIX: WATER				
PESTICIDES				
Gamma-BHC (Lindane)	8080	23	32-167	32-167
Heptachlor	8080	20	34-111	34-111
Aldrin	8080	21	42-122	42-122
Dieldrin	8080	38	36-146	36-146
Endrin	8080	37	30-147	30-147
4,4'-DDT	8080	36	25-160	25-160

MS/MSD CONSTITUENT	METHOD	MS/MSD PRECISION (RPD)	ACCURACY (% RECOVERY)	
			LCS	MS/MSD
MATRIX: SOILS/SEDIMENTS				
PESTICIDES				
Gamma-BHC (Lindane)	8080	29	D-116	D-116
Heptachlor	8080	28	D-136	D-136
Aldrin	8080	25	D-115	D-115
Dieldrin	8080	42	D-167	D-167
Endrin	8080	42	D-174	D-174
4,4'-DDT	8080	39	D-171	D171

TABLE 11

Richards-Gebaur AFB, MO
ACCURACY OBJECTIVES FOR METALS
MS/LCS RECOVERIES

TABLE 11

ACCURACY OBJECTIVES FOR METALS MS/LCS RECOVERIES IN SOIL/SEDIMENT DEVELOPMENT OF QUALITY PROGRAM PLAN RICHARDS-GEBAUR AFB, MO

METAL	SOIL MS/LCS RECOVERY LIMIT	WATERS
Antimony	66-115	68-121
Arsenic	60-126	63-131
Barium	56-132	64-124
Beryllium	60-109	72-121
Cadmium	54-112	58-128
Chromium	51-122	59-125
Cobalt	75-125	75-125
Copper	62-110	54-123
Lead	59-120	58-124
Mercury	75-128	93-117
Nickel	63-108	66-120
Selenium	60-126	73-124
Silver	59-118	63-122
Thallium	67-118	70-127
Tin	75-125	75-125
Vanadium	84-106	77-124
Zinc	50-123	69-118
Aluminum	75-125	65-121
Calcium	58-129	62-136
Iron	60-138	67-127
Manganese	75-125	70-115
Potassium	67-142	73-128
Sodium	70-126	57-138

TABLE 12

Richards-Gebaur AFB, MO
ACCURACY OBJECTIVES FOR LABORATORY MS/MSD, LCS RECOVERIES

TABLE 12

ACCURACY OBJECTIVES FOR (OTHER) LABORATORY MS/MSD, LCS RECOVERIES QUALITY ASSURANCE PROJECT PLAN DELIVERY ORDER 0001 RICHARDS-GEBAUR AFB, MO

ANALYTE	FREQUENCY	DQO LEVEL	AQUEOUS LIMIT	TYPE	SOIL/SEDIMENT LIMIT	TYPE
Geotechnical/Physical						
Grain size						
Density						
pH/eH						
Effective porosity						
Chemical						
Metals	1/10		See Table 11		See Table 11	
Volatiles by 8260	1/20		See Table 10		See Table 10	
Semivolatiles by 8270	1/20		See Table 10		See Table 10	
PCB/pesticides by 8080	1/20		See Table 10		See Table 10	
TDS	NA		NA		NA	
TSS	NA		NA		NA	
TOC	1/10		75-125		75-125	
TPH	1/20		78-109		70-129	

Notes: MS/MSD = Matrix spike/matrix spike duplicate.
MS = Matrix spike.
LCS = Laboratory control standard.
–, NA = Not applicable or available for this analyte.

TABLE 13

Richards-Gebaur AFB, MO
ACCURACY OBJECTIVES FOR SURROGATE RECOVERIES

Chemron, Inc.

TABLE 13

ACCURACY OBJECTIVES FOR SURROGATE RECOVERIES
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

ANALYTE GROUP METHOD	SURROGATE COMPOUND	AQUEOUS LIMIT		SOIL/SEDIMENT LIMIT	
		SAMPLES	LCS	SAMPLES	LCS
Volatiles by 8260	1,2-dichloroethane, d_4	87-115	87-115	83-122	83-122
	Toluene - d8	90-111	90-111	88-115	88-115
	4-bromofluorobenzene	80-114	80-114	75-127	75-127
Semivolatiles by 8270	Nitrobenzene - d5	35-110	35-110	23-120	23-120
	2-fluorobiphenyl	43-116	43-116	30-135	30-135
	Terphenyl-D14	33-141	33-141	18-137	18-137
	Phenol-D5	10-94	10-94	24-113	24-113
	2-fluorophenol	21-100	21-100	25-121	25-121
	2,4,6-tribromophenol	10-123	10-123	66-123	66-123
PCB/pesticides by 8080	TCMX	13-161		66-123	
	Dibutylchlorendate	d-165		18-176	

*Applies to both site samples and LCS.
TCMX = Tetrachloro m-xylene.

TABLE 14

Richards-Gebaur AFB, MO
SUMMARY OF ORGANIC AND INORGANIC ANALYTICAL PROCEDURES

Chemron, Inc.

TABLE 14 (continued)

SUMMARY OF NON-ANALYTICAL PROCEDURES

SOP Title
Corrective Action
Quality Control Procedures
Sample Handling
Data Handling
Instrument Calibration and Maintenance
Glassware/Container Cleaning

TABLE 15

PREVENTIVE MAINTENANCE CHECKLIST OF FIELD EQUIPMENT
QUALITY ASSURANCE PROJECT PLAN
DELIVERY ORDER 0001
RICHARDS-GEBAUR AFB, MO

EQUIPMENT	TASK	FREQUENCY
PID Photovac Microtip® with field calibration kit, 10.6 eV lamp and backup battery	Check battery	Daily
	Manual calibration	Daily/after each mesurement sequence
Horiba U-10 water quality checker	Maintain turbidity sensor	Daily or when needed
	Maintain conductivity sensor	Daily or when needed
	Recharge reference sensor (with reference solution)	Once every 2 months
	Replace faulty sensors • pH sensor • reference sensor • DO sensor	As needed
	Replace faulty probe (new probes must be manually calibrated for all four parameters)	As needed
Water level meter	Check battery	Daily and before each use

FIGURE 1

Richards-Gebaur AFB, MO
SITE LOCATION MAP

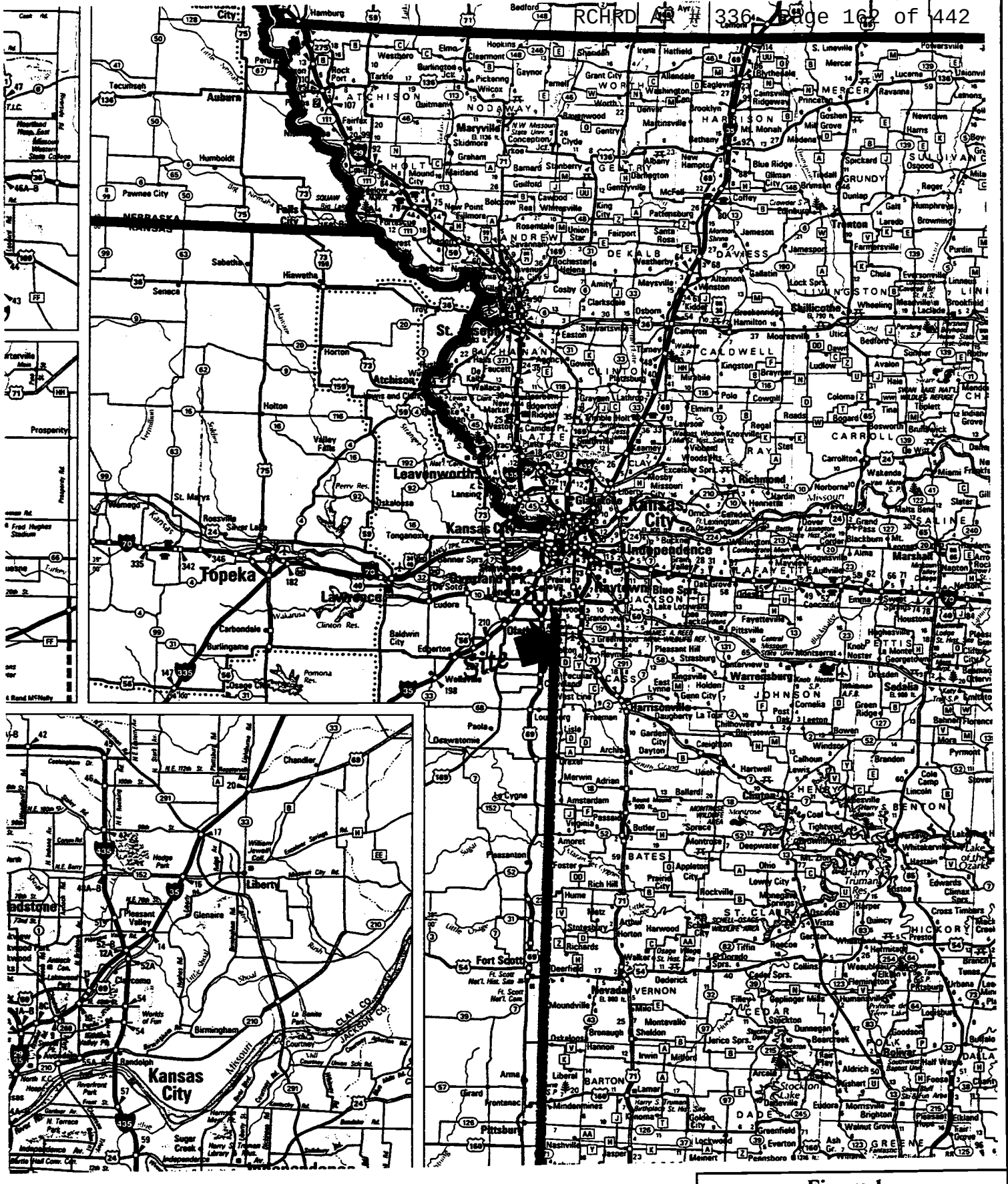


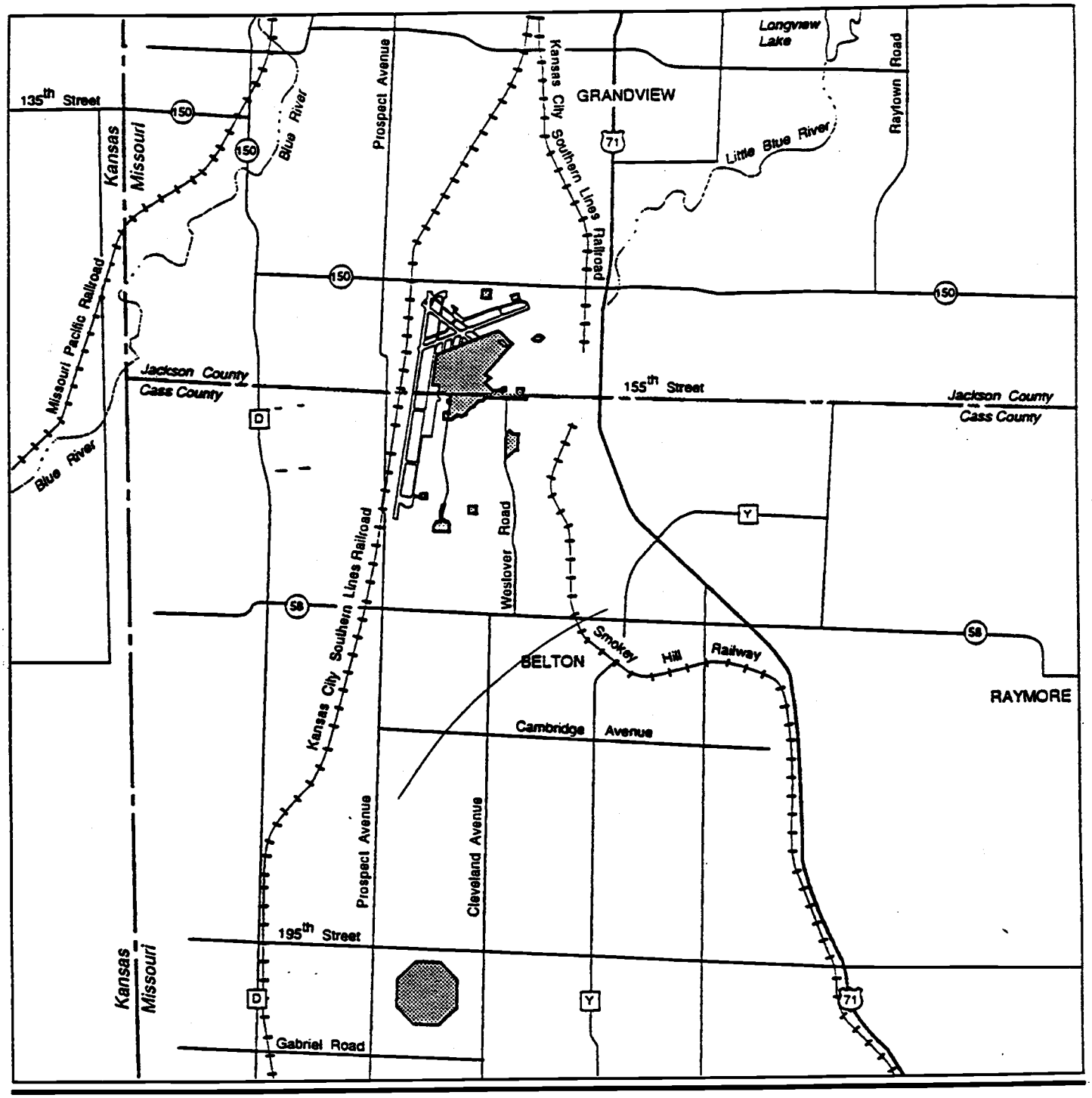
Figure 1
SITE LOCATION MAP

Richards-Gebaur Air Force Base
Belton, Missouri

Dames & Moore

FIGURE 2

Richards-Gebaur AFB, MO
SITE PLAN MAP



EXPLANATION

- Base Property
- U. S. Highway
- State Highway
- County Road

0 1750 3500 7000 Feet

N

Figure 2
SITE LOCATION MAP

Richard-Gebaur Air Force Base
Belton, Missouri

Dames & Moore

FIGURE 3

Richards-Gebaur AFB, MO
SAMPLING LOCATIONS

SAMPLE LOCATIONS WILL BE PROVIDED WITH
SITE SPECIFIC PLANS.

FIGURE 4

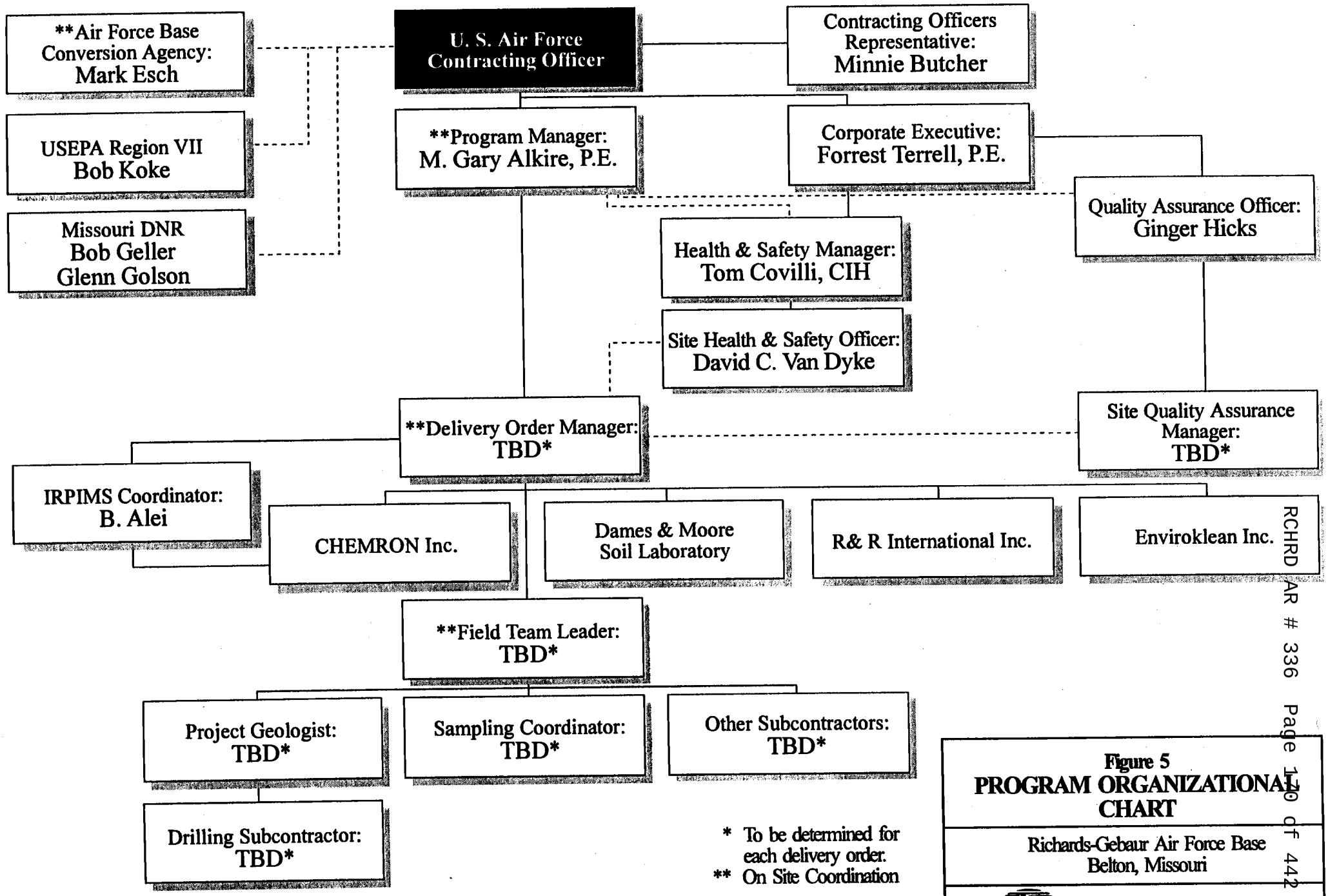
Richards-Gebaur AFB, MO
PROJECT SCHEDULE

PROJECT SCHEDULE WILL BE
PROVIDED WITH SITE SPECIFIC PLANS

FIGURE 5

Richards-Gebaur AFB, MO
PROJECT ORGANIZATION CHART

Organizational Chart Richards-Gebaur Air Force Base Delivery Order No. 1



* To be determined for each delivery order.
** On Site Coordination

Figure 5
PROGRAM ORGANIZATIONAL CHART
Richards-Gebaur Air Force Base
Belton, Missouri
DAMES & MOORE

FIGURE 6

Richards-Gebaur AFB, MO
CHEMRON ENVIRONMENTAL LABORATORIES
ORGANIZATION CHART

CHEMRON ORGANIZATIONAL CHART

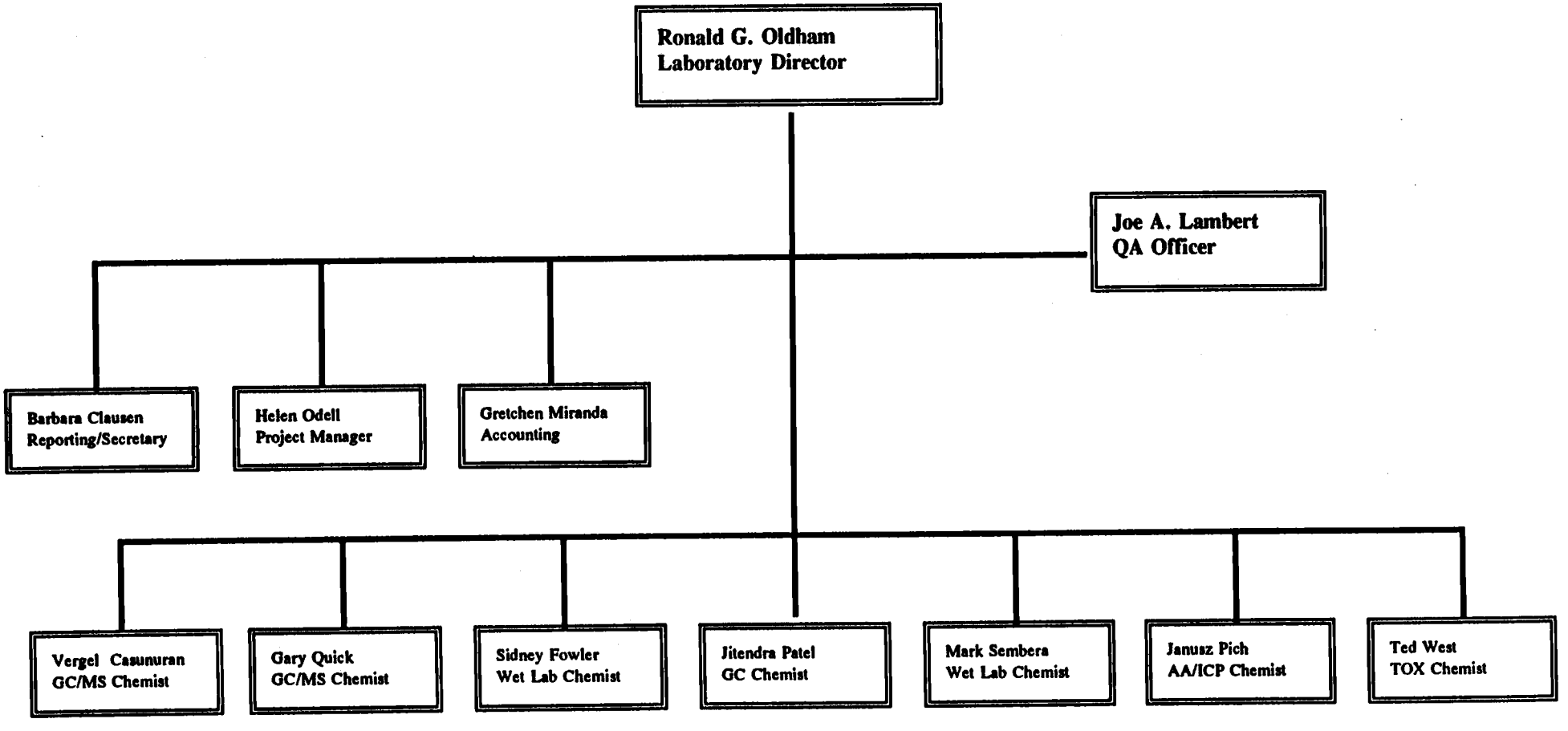


FIGURE 7

Dames & Moore
SAMPLE JAR LABEL

DAMES & MOORE | PROJECT #

SAMPLE I.D.

PRESERVATIVES: (CIRCLE ONE)

1. HNO_3 2. HCl 3. H_2SO_4 4. UNPRESERVED

5. Hexane Rinsed 6. TEFLON CAPS 7. Other_____

ANALYSIS | DATE

Enlarged Label Format

Figure 7
Dames & Moore
Sample Jar Label

FIGURE 8

Dames & Moore
CHAIN-OF-CUSTODY FORM

DAMES & MOORE

Turnaround Time

☐ Rush (preapproved by Lab)

☐ Normal

SHIPPING DETAILS:

Method of Shipment _____

Contents Temperature _____ C

Comments _____

PROJECT NAME: _____

PROJECT #: _____

Send Results To:

PROJECT MANAGER: _____

LAB USE ONLY	DATE	CONTAINERS	No.	SAMPLE ID	SAMPLE TYPE	ANALYSIS REQUESTED	REMARKS/PRESERVATIVES

CHAIN OF CUSTODY RECORD

COMMENTS

SAMPLER: (SIGNATURE)	DATE

RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE)
RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE)

RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE)	
RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED FOR LABORATORY: BY: (SIGNATURE)	DATE/TIME

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Figure 1
Dames & Moore
Chain of Custody Form

FIGURE 9

EQUIPMENT CALIBRATION LOG

EQUIPMENT CALIBRATION LOG[a]

Page_____ of_____

Instrument Name_____

Manufacturer_____

Equipment Serial No._____ D&M Asset No._____

Date Acquired or placed in Service_____

Calibration Record

Date_____ Calibrated By_____

Comments:_____

_____

_____

New Calibration Due_____

Calibration Record

Date_____ Calibrated By_____

Comments:_____

_____

_____

New Calibration Due_____

Calibration Record

Date_____ Calibrated By_____

Comments:_____

_____

_____

New Calibration Due_____

---

[a]Several pages will be required for each instrument.

FIGURE 9
Equipment Calibration Log

FIGURE 10

SURVEILLANCE REPORT

SURVEILLANCE REPORT

Date: _____

Project: _____

Project Location: _____

Project No.: _____

Purpose of Survey: _____

_____

Activities Surveyed: _____

_____

Location of Activities : _____

_____

Personnel Contacted: _____

_____

	Date	Conducted By
Preceding Survey:	_____	_____
This Survey:	_____	_____

List of Reference Documents Used: _____

_____

_____

_____

_____

FIGURE 10
Surveillance Report

Observations Noted/Action Recommended:

Findings Noted/Action Recommended:

Nonconformances Noted/Action Recommended:

General Comments/Action Taken:

Submitted By: _____
(Signature)

_____
(Name)

_____
(Title)

Reviewed By: _____
(Signature)

_____
(Name)

_____
(Title)

FIGURE 11

EXAMPLE AUDIT FORM

AUDIT FINDING SHEET

FINDING #_____

Job No._____
Project_____
Page_____of____

Subject:	Audit Date(s):
Audit Participants:	Responsible Party(s):
Finding:	
Checklist Reference:	
Recommended Action:	Auditor:_____ Date::_____ Acknowledged By: _____ Date:_____
Response For Close Out:	

FIGURE 11
Example Audit Form

FIGURE 12

EXAMPLE AUDIT REPORT FORMAT

AUDIT REPORT FORMAT

Lead-in Statement (Describe purpose of report)

Breakdown of Events

I. Purpose of Audit
II. Scope
III. Audit Basis
IV. Time and Place
V. Personnel Contacted
VI. D&M Audit Team Members
VII. Summary of events

Initial Meeting (e.g., comments on changes in scope, etc.)

Physical Audit (e.g., reference the checklist used, etc.)

Exit Interview & Conclusion (e.g., indicate positive and negative findings)

VIII. A. Findings and Recommendations

1) Positive Findings
(i.e., discuss strong points encountered, areas that were being implemented well, etc.).

2) Negative Findings
(i.e., discuss nature of findings, reference attatched finding sheets, and discuss recommended remedies and/or corrective actions).

B. Observations
(Discuss each in detail)

IX. Evaluation

X. Follow-up
Identify parties responsible for follow-up. Indicate when remedies and/or corrective actions will be completed. Discuss whether a reaudit will be required.

FIGURE 12
Example Audit Report Format

CONTRACT QUALITY ASSURANCE PROJECT PLAN
RICHARDS-GEBAUR AIR FORCE BASE

F41624-94-D-8102

DELIVERY ORDER 0001

AIR FORCE CENTER FOR ENVIRONMENTAL EXCELLENCE

BROOKS A.F.B., TEXAS

CHEMRON LAB STANDARDS OPERATING PROCEDURES

TABLE OF CONTENTS

CHEMRON INC. - STANDARD OPERATING PROCEDURE 3005

ACID DIGESTION OF WATERS FOR TOTAL RECOVERABLE OR DISSOLVED METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 3005 is a acid digestion procedure used to prepare surface water and ground water samples for analysis by flame atomic absorption spectroscopy (FLAA) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by SOP 3005 may be analyzed by FLAA or ICP for the following metals:

Aluminum	Magnesium
Antimony	Manganese
* Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	* Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

* ICP only

1.2 For the analysis of total dissolved metals, the sample is filtered at the time of collection, prior to acidification with nitric acid.

2.0 SUMMARY OF METHOD

2.1 Total recoverable metals: The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

2.2 Dissolved metal: The sample is filtered through a 0.5 um filter at the time of collection and the liquid phase is then acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is again filtered (if necessary) and diluted to volume and is then ready for analysis.

3.0 INTERFERENCES

3.1 The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers of assorted sizes.

4.2 Watch glasses.

4.3 Qualitative filter paper and filter funnels.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine level of impurities. If method blank is <MDL, then acid can be used.

5.3 Concentrated hydrochloric acid, reagent grade (HCl): Acid should be analyzed to determine level of impurities. If method blank is <MDL, then acid can be used.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses all relevant sampling considerations.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Sampling:

6.3.1 **Total recoverable metals:** All samples must be acidified at the time of collection with HNO_3 (5 mL/L).

6.3.2 **Dissolved metals:** All samples must be filtered through a 0.5 um filter and then acidified at the time of collection with HNO_3 (5 mL/L).

7.0 PROCEDURE

7.1 Transfer a 100-mL aliquot of well mixed sample to a beaker.

7.2 For metals that are to be analyzed by FLAA or ICP, add 2 mL of concentrated HNO_3 and 5 mL of concentrated HCl. The sample is covered with a ribbed watch glass and heated on a steam bath or hot plate at 90 to 95°C until the volume has been reduced to 15-20 mL.

CAUTION: Do not boil. Antimony is easily lost by volatilization from hydrochloric acid media.

7.3 Remove the beaker and allow to cool. Wash down the beaker walls and watch glass with Type II water and, when necessary, filter or centrifuge the nebulizer. Filtration should be done only if there is concern that insoluble materials may clog the nebulizer; this additional step is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and prerinsed with dilute HNO_3.

7.4 Adjust the final volume to 100 mL with Type II water.

8.0 QUALITY CONTROL

8.1 For each analytical batch of samples processed, blanks (Type II water and reagents) should be carried throughout the entire sample preparation and analytical process. The blank will be used to determine if the samples are being contaminated.

8.2 Duplicate samples should be processed on a routine basis. A duplicate sample is a sample brought through the whole sample preparation and analytical process. Duplicate samples will be used to determine precision, and should be analyzed on a 10% basis.

8.3 Spiked samples or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each group of samples processed or every 10 samples whichever is greater.

Bibliography

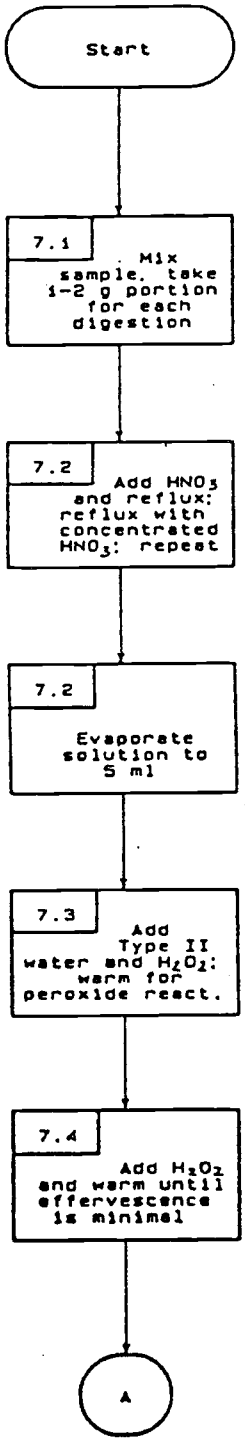
1. U. S. Environmental Protection Agency, "Test Methods for Evaluating Solid Waste, SW-846", November 1986.

Revision - 1.2
Date - December 6, 1994

Approved: _____ Date: 12/06/94

METHOD 3050

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS



CHEMRON INC. - STANDARD OPERATING PROCEDURE 3050

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 3050 is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA respectively) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also Paragraph 2.1):

FLAA		GFAA
Aluminum	Magnesium	Arsenic
Barium	Manganese	Beryllium
Beryllium	Molybdenum	Cadmium
Cadmium	Nickel	Chromium
Calcium	Potassium	Cobalt
Chromium	Sodium	Iron
Cobalt	Thallium	Molybdenum
Copper	Vanadium	Selenium
Iron	Zinc	Thallium
Lead		Vanadium

2.0 SUMMARY OF METHOD

2.1 A representative 1- to 2-g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, and (2) the flame AA or ICP analysis of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Mo, Pb, Ni, K, Na, Tl, V, and Zn. Dilute nitric acid is employed as the final dilution acid for the furnace AA analysis of AS, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V. A separate sample shall be dried for a total solids determination if required.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method 3050 is applicable to a given waste.

4.0 APPARATUS AND MATERIALS

4.1 Conical Phillips beakers: 250 ml.
4.2 Watch glasses.
4.3 Drying ovens: That can be maintained at 30°C.
4.4 Thermometer: That covers range of 0 to 200°C.
4.5 Whatman No. 41 filter paper (or equivalent).
4.6 Centrifuge and centrifuge tubes.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.

5.3 Concentrated hydrochloric acid, reagent grade (HCL): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.

5.4 Hydrogen peroxide (30%) (H_2O_2): Oxidant should be analyzed to determine level of impurities.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses all of the relevant sampling considerations.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Nonaqueous samples shall be refrigerated upon receipt and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.01 g and transfer to a conical beaker a 1.00- to 2.00-g portion of sample.

7.2 Add 10 mL of 1:1 of HNO_3, mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 to 15 min without boiling.

Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

7.3 After Step 7.2 has been completed and the sample has cooled, add 2 mL of Type II water and 3 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

7.4 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

7.5 If the sample is be prepared for (a) the ICP analysis of AS and Se, or (b) the flame AA or ICP analysis of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Na, Tl, V, and Zn, then add 5 mL of concentrated HCl and 10 mL of Type II water, return the covered beaker to the hot plate, and reflux for an additional 15 min without boiling. After cooling, dilute to 100 mL with Type II water. Particulates in the digestate that may clog the nebulizer should be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.5.1 **Filtration:** Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with Type II water.

7.5.2 **Centrifugation:** Centrifugation at 2,000-3,000 rpm for 10 min is usually sufficient to clear the supernatant.

7.5.3 The diluted sample has an approximate acid concentration of 5.0% (v/v) HCl and 5.0% (v/v) HNO_3. The sample is now ready for analysis.

7.6 If the sample is being prepared for the furnace analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl and V cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL. After cooling, dilute to 100 mL with Type II water. Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.6.1 **Filtration:** Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with Type II water.

7.6.2 **Centrifugation:** Centrifugation at 2,000-3,000 rpm for 10 min is usually sufficient to clear the supernatant.

7.6.3 The diluted digestate solution contains approximately 5% (v/v) HNO_3. For analysis, withdraw aliquots of appropriate volume and add any required reagent or matrix modifier. The sample is now ready for analysis.

7.7 Calculations:

7.7.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

7.7.2 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

8.0 QUALITY CONTROL

8.1 For each group of samples processed, preparation blanks (Type II water and reagents) should be carried throughout the entire sample preparation and analytical process. The blank will be used to determine if the samples are being contaminated.

8.2 Duplicate samples should be processed on a routine basis. Duplicate samples will be used to determine precision, and should be analyzed on a 10% basis.

8.3 Spiked samples or standard reference materials must be employed to determine accuracy. A spiked sample should be included with each group of samples processed or every 10 samples whichever is greater.

8.4 The concentration of all calibration standards should be verified against a quality control check sample obtained from an outside source.

Bibliography

1. U. S. Environmental Protection Agency, "Test Methods for Evaluating Solid Waste, SW-846", November 1986.

Revision - 1.2
Date - December 6, 1994

Approved: _____ Date: 12/06/94

CHEMRON INC. - STANDARD OPERATING PROCEDURE 418.1

PETROLEUM HYDROCARBONS, TOTAL RECOVERABLE
(SPECTROPHOTOMETRIC, INFRARED)

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP 418.1) is for the measurement of fluorocarbon-113 extractable petroleum hydrocarbons from surface and saline waters, industrial and domestic wastes.

1.2 This SOP is applicable to measurement of lights fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.

1.3 This method is sensitive to levels of 1 mg/1 and less ,and may be extended to ambient monitoring.

2.0 SUMMARY OF METHOD

2.1 The sample is acidified to a low pH (<2) and serially extracted with fluorocarbon-113 in a separatory funnel. Interferences are removed with silica gel adsorbent. Infrared analysis of the extract is performed by direct comparison with standards.

3.0 DEFINITIONS

3.1 As in the case of Oil and Grease, the parameter of Petroleum Hydrocarbons is defined by the method. The measurement may be subject to interferences and the results should be evaluated accordingly.

3.2 Oil and Grease is a measure of biodegradable animal greases and vegetable oils along with the relative non-biodegradable mineral oils. Petroleum hydrocarbons is the measure of only the mineral oils. Maximum information may be obtained using both methods to measure and characterize oil and grease of all sources.

4.0 SAMPLING AND STORAGE

4.1 A representative sample of 1 liter volume should be collected in a glass bottle. Because losses of grease will occur

on sampling equipment, the collection of a composite sample is impractical. The entire sample is consumed by this test; no other analyses may be performed using aliquots of the sample.

4.2 A delay between sampling and analysis of greater than 4 hours requires sample preservation by the addition of 5 mL HCL (6:1). A delay of greater than 48 hrs also requires refrigeration for sample preservation.

5.0 APPARATUS

5.1 Separatory funnel: 2,000 mL, with Teflon stopcock.

5.2 Filter paper: Whatman No. 40, 11 cm.

5.3 Infrared spectrophotometer: Scanning or fixed wavelength, for measurement around 2,950 cm.

5.4 Cells: 10 mm, 50 mm, and 100 mm pathlength, sodium chloride or infrared grade glass.

5.5 Magnetic stirrer: With Teflon coated stirring bars.

6.0 REAGENTS

6.1 Hydrochloric acid, 1:1. Mix equal volumes of conc HCl and distilled water.

6.2 Fluorocarbon-113,(1,2,2-trichloro-1,2,2-trifluoroethane), b.p. 48°C.

6.3 Sodium sulfate, anhydrous crystal.

6.4 Silica gel, 60-200 mesh, Davidson Grade 950 or equivalent. Should contain 1-2% water as defined by residue test at 130°C. Adjust by overnight equilibration if needed.

6.5 Calibration mixtures:

6.5.1 Reference oil: Pipet 15.0 mL n-hexadecane, 15.0 mL isooctane, and 10.0 mL chlorobenzene into a 50 mL glass stoppered bottle. Maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

6.5.2 Stock standard: Pipet 1.0 mL reference oil (6.5.1) into a tared 200 mL volumetric flask and immediately stopper. Weigh and dilute to volume with fluorocarbon-113.

6.5.3 Working standards: Pipet appropriate volumes of stock standard (6.5.2) into 100 mL volumetric flasks according to the cell pathlength to be used. Dilute to volume with fluorocarbon-113. Calculate concentration of standards from the stock standard.

7.0 PROCEDURE

7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 2 mL hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.

7.2 Pour the sample into a separatory funnel.

7.3 Add 30 mL fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate.

7.4 Filter the solvent layer through a funnel containing solvent-moistened filter paper into a 100 mL volumetric flak.

NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

7.5 Repeat (7.3 and 7.4) twice more with 30 mL portions of fresh solvent, combining all solvent into the volumetric flask.

7.6 Rinse the tip of the separatory funnel, filter paper, and the funnel with a total of 5-10 mL solvent and collect the rinsings in the flask. Dilute the extract to 100 mL. If the extract is known to contain greater than 100 mg of non-hydrocarbon organic material, pipet an appropriate portion of the sample to a 100 mL volumetric and dilute to volume.

7.7 Discard about 5-10 mL solution from the volumetric flask. Add 3 g silica gel (6.4) and a stirring bar; stopper the volumetric flask, and stir the solution for a minimum of 5 min on a magnetic stirrer.

7.8 Select appropriate working standards and cell pathlength according to the following table of approximate working ranges:

Pathlength	Range
10 mm	2-40 mg
50 mm	0.5-8 mg
100 mm	0.1-4 mg

Calibrate the instrument for the appropriate cells using a series of working standards (6.5.3). If is not necessary to add silica gel to the standards. Determine absorbance directly for each solution at the absorbance maximum at about 2930 cm^{-1}. Prepare a calibration plot of absorbance vs. mg petroleum hydrocarbons per 100 mL solution.

7.9 After the silica gel has settled in the sample extract, fill a clean cell with solution and determine the absorbance of the extract. If the absorbance exceeds 0.8 prepare an appropriate dilution.

NOTE: The possibility that the absorptive capacity of the silica gel has been exceeded can be tested at this point by adding another 3.0 g silica gel to the extract and repeating the treatment and determination.

7.10 Determine the concentration of petroleum hydrocarbons in the extract by comparing the response against the calibration plot.

8.0 CALCULATIONS

8.1 Calculate the petroleum hydrocarbons in the sample using the formula:

$$mg/1\ PetroleumHydrocarbons = \frac{R \times D}{V}$$

Where:

R = mg of Petroleum Hydrocarbons as determined from the calibration plot (7.10).
D = Extract dilution factor, if used.
V = Volume of sample, in liters.

Bibliography

1. "Methods for Chemical Analysis of Water and Wastes," EPA/600/4-79-020, March 1983.
2. Chemron Standard Operating Procedure 418.1, March 1983.

Rev. 2.0
Date - December 7, 1994

Approved: _____ Date: 12/07/94

CHEMRON INC. - STANDARD OPERATING PROCEDURE 6010

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 6010 determines elements including metals in solution. The SOP is applicable to a large number of metals and wastes. All matrices, including ground water, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes, undergo digestion prior to analysis.

1.2 Elements for which SOP 6010 is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Table 1 provide concentration ranges for clean aqueous samples. Chemron restricts use of this method to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 The method of standard addition (MSA) (Paragraph 8.5.3) is used for the analysis of all EP extracts and sample digests unless either serial dilution or matrix spike addition demonstrates that it is not required.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples are solubilized or digested using appropriate Sample Preparation Methods (e.g. SOP's 3005-3050).

2.2 SOP 6010 describes the simultaneous, or sequential, multielemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is performed for trace element determination. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not performed in cases of line broadening where a background correction measurement would actually degrade the analytical result. Tests for the presence of additional interferants are described in Section 8.5.

TABLE 1. RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength[a] (nm)	Estimated Detection Limit[b] (ug/L)
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	See note c
Selenium	196.026	75
Silicon	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[a]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Paragraph 3.1). In time, other elements may be added as more information becomes available and as required.

[b]The estimated instrumental detection limits shown are taken from Reference 1 in Section 10.0 below. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c]Highly dependent on operating conditions and plasma position.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line. Potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents is assumed.

3.1.1 The interference is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

3.1.2 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

3.1.3 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they are reduced by diluting the sample, by using a peristaltic pump or by using the standard additions method. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem is controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

TABLE 2. ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL

Analyte	Wavelength (nm)	Interferent [a,b] Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	—	—	—	—	—	—	0.21	—	—	1.4
Antimony	206.833	0.47	—	2.9	—	0.08	—	—	—	0.25	0.45
Arsenic	193.696	1.3	—	0.44	—	—	—	—	—	—	1.1
Barium	455.403	—	—	—	—	—	—	—	—	—	—
Beryllium	313.042	—	—	—	—	—	—	—	—	0.04	0.05
Boron	249.773	0.04	—	—	—	0.32	—	—	—	—	—
Cadmium	226.502	—	—	—	—	0.03	—	—	0.02	—	—
Calcium	317.933	—	—	0.08	—	0.01	0.01	0.04	—	0.03	0.03
Chromium	267.716	—	—	—	—	0.003	—	0.04	—	—	0.04
Cobalt	228.616	—	—	0.03	—	0.005	—	—	0.03	0.15	—
Copper	324.754	—	—	—	—	0.003	—	—	—	0.05	0.02
Iron	259.940	—	—	—	—	—	—	0.12	—	—	—
Lead	220.353	0.17	—	—	—	—	—	—	—	—	—
Magnesium	279.079	—	0.02	0.11	—	0.13	—	0.25	—	0.07	0.12
Manganese	257.610	0.005	—	0.01	—	0.002	0.002	—	—	—	—
Molybdenum	202.030	0.05	—	—	—	0.03	—	—	—	—	—
Nickel	231.604	—	—	—	—	—	—	—	—	—	—
Selenium	196.026	0.23	—	—	—	0.09	—	—	—	—	—
Silicon	288.158	—	—	0.07	—	—	—	—	—	—	0.01
Sodium	588.995	—	—	—	—	—	—	—	—	0.08	—
Thallium	190.864	0.30	—	—	—	—	—	—	—	—	—
Vanadium	292.402	—	—	0.05	—	0.005	—	—	—	0.02	—
Zinc	213.856	—	—	—	0.14	—	—	—	0.29	—	—

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L,
Ca - 1000 mg/L,
Cr - 200 mg/L,
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L,
Mn - 200 mg/L,
Tl - 200 mg/L,
V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 JY-70 simultaneous and sequential plasma emission spectrometer.

4.1.2 Argon gas supply: Welding grade or better.

4.2 Operating conditions: The analyst follows the instruction provided by the instrument's manufacturer. For operation with organic solvents, the analyst may use the auxiliary argon inlet, as well as solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects are established for each individual analyte line on that particular instrument. All measurements must be within instrument linear range where coordination factors are valid. The analyst (1) verifies that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintains quality control data confirming instrument performance and analytical results.

5.0 REAGENTS

5.1 Acids used in the preparation of standards and for sample processing are reagent grade or better. Redistilled acids may be used.

5.1.1 Concentrated hydrochloric acid (HCL).

5.1.2 Hydrochloric acid (1:1): Analyst adds 500 mL concentrated HCl to 400 mL Type II water and dilute to 1 liter.

5.1.3 Concentrated nitric acid (HNO_3).

5.1.4 Nitric acid (1:1): Analyst adds 500 mL concentrated HNO_3 to 400 mL Type II water and dilute to 1 liter.

5.2 Type II water (ASTM D1193): Water is monitored for impurities.

5.3 Standard stock solutions may be purchased or prepared from ultra-high purity grade chemicals or metals (99.99 to 99.999% pure). All salts are dried for 1 hr at 105°C, unless otherwise specified.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the mole fraction and the weight of the metal salt added.

Metal

$$concentration\ (ppm) = \frac{weight\ (mg)}{volume\ (L)}$$

Metal salts

$$concentration\ (ppm) = \frac{weight\ (mg)\ x\ mole\ fraction}{volume\ (L)}$$

5.3.1 **Aluminum solution**, stock, 1 mL == 100 µg Al: Analyst dissolves 0.10 g of aluminum metal, weighed accurately to at least four significant figures, in an acid mixture of 4 mL of (1:1) HCl and 1 mL of concentrated HNO_3 in a beaker. Analyst warms gently to effect solution. When solution is complete, it is transfered quantitatively to a liter flask, and an additional 10 mL of (1:1) HCl is added. Solution is then diluted to 1,000 mL with Type II water.

5.3.2 **Antimony solution**, stock, 1 mL = 100 µg Sb: Analyst dissolves 0.27 g $K(SbO)C_4H_4O_6$ (mole fraction Sb = 0.3749), weighed accurately to at least four significant figures, in Type II water, adds 10 mL (1:1) HCl, and dilutes to 1,000 mL with Type II water.

5.3.3 **Arsenic solution**, stock, 1 mL = 100 µg As: Analyst dissolves 0.13 g of As_2O_3 (mole fraction 0.7574), weighed accurately to at least four significant figures, in 100 mL of Type II water containing 0.4 g NaOH, acidifies the solution with 2 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.4 **Barium solution**, stock, 1 mL = 100 µg Ba: Analyst dissolves 0.15 g $BaCl_2$ (mole fraction Ba = 0.6596), dried at 250°C for 2 hr, weighed accurately to at least four significant figures, in 10 mL Type II water with 1 mL (1:1) HCl, adds 10.0 mL (1:1) HCl and dilutes to 1,000 mL with Type II water.

5.3.5 **Beryllium solution**, stock, 1 mL = 100 µg Be: Do not dry. Analyst dissolves 1.97 g $BeSO_4*4H_2O$ (mole fraction Be = 0.0509), weighed accurately to at least four significant figures, in Type II water, adds 10.0 mL concentrated HNO_3, and dilutes to 1,000 mL with Type II water. Mole fraction = 0.0509.

5.3.6 **Boron solution**, stock 1 mL = 100 µg B: Do not dry. Analyst dissolves 0.57 g anhydrous H_3BO_3 (mole fraction B = 0.1748), weighed accurately to at least four significant figures, in Type II water and dilutes to 1,000 mL. Using a reagent meeting ACS specifications, the bottle is kept tightly stoppered, and stored in a desiccator to prevent the entrance of atmospheric moisture.

5.3.7 **Cadmium solution**, stock, 1 mL = 100 µg Cd: Analyst dissolves 0.11 g Cd (mole fraction Cd = 0.8754), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Analyst heats to increase rate of dissolution, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL Type II water.

5.3.8 **Calcium solution**, stock, 1 mL = 100 µg Ca: Analyst suspends 0.25 g $CaCO_3$ (mole Ca fraction = 0.4005), dried at 180°C for 1 hr before weighing, weighed accurately to at least four significant figures, in Type II water and dissolves cautiously with a minimum amount of (1:1) HNO_3. Analyst then adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.9 **Chromium solution**, stock, 1 mL = 100 µg Cr: Analyst dissolves 0.19 g CrO_3 (mole fraction Cr = 0.5200), weighed accurately to at least four significant figures, in Type II water. When solution is complete, it is acidified with 10 mL concentrated HNO_3 and diluted to 1,000 mL with Type II water.

5.3.10 **Cobalt solution**, stock, 1 mL = 100 µg Co: Analyst dissolves 0.1000 g Cobalt metal, weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3, adds 10.0 mL (1:1) concentrated HCl and dilutes to 1,000 mL Type II water.

5.3.11 **Copper solution**, stock, 1 mL = 100 µg Cu: Analyst dissolves 0.13 g CuO (mole fraction Cu = 0.7989), weighed accurately to at least four significant figures), in a minimum amount of (1:1) HNO_3, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.12 **Iron solution**, stock, 1 mL = 100 µg Fe: Analyst dissolves 0.14 g Fe_2O_3 (mole fraction Fe = 0.6994), weighed accurately to at least four significant figures, in a warm mixture of 20 mL (1:1) HCl and 2 mL of concentrated HNO_3, and dilutes to 1,000 mL with Type II water.

5.3.13 **Lead solution**, stock, 1 mL = 100 µg Pb: Analyst dissolves 0.16 g $Pb(NO_3)_2$ (mole fraction Pb = 0.6256), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3, adds 10 mL (1:1) HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.14 **Magnesium solution**, stock, 1 mL = 100 µg Mg: Analyst dissolves 0.17 g MgO (mole fraction Mg = 0.6030), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3, adds 10.0 mL (1:1) concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.15 **Manganese solution**, stock, 1 mL = 100 µg Mg: Analyst dissolves 0.1000 g of manganese metal, weighed accurately to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO_3) and dilutes to 1,000 mL with Type II water.

5.3.16 **Molybdenum solution**, stock, 1 mL = 100 µg Mo: Analyst dissolves 0.20 g $(NH_4)_6Mo_7O_{24}*4H_2O$ (mole fraction Mo = 0.5772), weighed accurately to at least four significant figures, in Type II water and dilutes to 1,000 mL with Type II water.

5.3.17 **Nickel solution**, stock, 1 mL = 100 µg Ni: Analyst dissolves 0.1000 g of nickel metal, weighed accurately to at least four significant figures, in 10.0 mL hot concentrated HNO_3, cool, and dilutes to 1,000 mL with Type II water.

5.3.18 **Potassium solution**, stock, 1 mL = 100 µg K: Analyst dissolves 0.19 g KCl (mole fraction K = 0.5244) dried at 110°C, weighed accurately to at least four significant figures, in Type II water and dilutes to 1,000 mL.

5.3.19 **Selenium solution**, stock, 1 mL = 100 µg Se: Do not dry. Analyst dissolves 0.17 g H_2SeO_3 (mole fraction Se = 0.6123), weighed accurately to at least four significant figures, in Type II water and dilutes to 1,000 mL.

5.3.20 **Silica solution**, stock, 1 mL = 100 µg SiO_2: Do not dry. Analyst dissolves 0.47 g $Na_2SiO_3*9H_2O$ (mole fraction Si = 0.09884), weighed accurately to at least four significant figures, in Type II water, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.21 **Silver solution**, stock, 1 mL = 100 µg Ag: Analyst dissolves 0.25 g $AgNO_3$ (mole fraction Ag = 0.6350), weighed accurately to at least four significant figures, in Type II water and 10 mL concentrated HNO_3, and dilutes to 1,000 mL with Type II water.

5.3.22 **Sodium solution**, stock, 1 mL = 100 µg Na: Analyst dissolves 0.25 g NaCl (mole fraction Na = 0.3934), weighed accurately to at least four significant figures, in Type II water, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.23 **Thallium solution**, stock, 1 mL = 100 µg Tl: Analyst dissolves 0.13 g $TiNO_3$ (mole fraction Ti = 0.7672), weighed accurately to at least four significant figures, in Type II water, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.24 **Vanadium solution**, stock, 1 mL = 100 µg V: Analyst dissolves 0.23 g NH_4VO_3 (mole fraction V = 0.4356), weighed accurately to at least four significant figures, in a minimum amount of concentrated HNO_3, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.3.25 **Zinc solution**, stock, 1 mL = 100 µg Zn: Analyst dissolves 0.12 g ZnO (mole fraction Zn = 0.8034), weighed accurately to at least four significant figures, in a minimum amount of dilute HNO_3, adds 10.0 mL concentrated HNO_3 and dilutes to 1,000 mL with Type II water.

5.4 Mixed calibration standard solutions: Mixed calibration standard solutions are prepared in volumetric flasks (see Table 3). Analyst adds 2 mL (1:1) HNO_3 and 10 mL of (1:1) HCl and dilutes to 100 mL with Type II water (see NOTE, below). Prior to preparing the mixed standards, each stock solution is analyzed separately to determine possible spectral interference or the presence of impurities. Care is taken when preparing the mixed standards to ensure that the elements are compatible and stable together. The mixed standard solutions are then transfered to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards are prepared, as needed, with the realization that concentration can change on aging. Calibration standards are initially verified using a quality control sample (see Paragraph 5.8) and monitored weekly for stability. Some typical calibration standard combinations are listed in Table 3. All mixtures are then scanned using a sequential spectrometer to verify the absence of interelement spectral interference in the recommended mixed standard solutions.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, the analyst adds 15 mL of Type II water and warms the flask until the solution clears. It is then cooled and diluted to 100 mL with Type II water. For this acid combination, the silver concentration is limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

5.5 Two types of blanks are performed for the analysis. The calibration blank is used in establishing the analytical curve, and the reagent blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by diluting 2 mL of (1:1) HNO_3 and 10 mL of (1:1) HCl to 100 mL with Type II water. A sufficient quantity is prepared to flush the system between standards and samples.

5.5.2 The reagent blank contains all the reagents and in the same volumes as used in the processing of the samples. The reagent blank is carried through the complete procedure and contains the same acid concentration in the final solution as the sample solution used for analysis.

5.6 The instrument check standard is prepared by the analyst by combining compatible elements at concentrations equivalent to the midpoint of their respective calibration curves (see Paragraph 8.6.2.1 for use).

5.7 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The analyst spikes the sample with the elements of interest at approximate concentration of 10 times the instrumental detection limits. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure is not necessary.

5.8 The quality control sample is prepared in the same acid matrix as the calibration standards at 10 times the instrumental detection limits and in accordance with the instructions provided by the supplier.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chemron SOP 1000.

TABLE 3. MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo, and Si
IV	Al, Ca, Cr, K, Na, and Ni
V	Ag (see Note to Paragraph 5.4), B, Mg, Sb, and Tl

7.0 PROCEDURE

7.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005-3050). The method of standard addition (MSA) (Paragraph 8.5.3) is used for the analysis of all EP extracts and sample digests unless either serial dilution or matrix spike addition demonstrates that it is not required. An internal standard may be substituted for the MSA.

7.2 The instrument is set up with proper operating parameters established in Paragraph 4.2. The instrument is allowed to become thermally stable before beginning (usually requiring at least 30 min of operation prior to calibration).

7.3 The instrument is then profiled and calibrated according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Paragraph 5.4. The analyst flushes the system with the calibration blank (5.5.1) between each standard (see NOTE, below). (Using the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.)

NOTE: For boron concentration greater than 500 ug/L, extended flush times of 1 or 2 min may be required.

7.4 Before beginning the sample run, the analyst reanalyzes the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5% (or the established control limits, whichever is lower). If they do, the analyst follows the recommendations of the instrument manufacturer to correct for this condition.

7.5 Next, the analyst flushes the system with the calibration blank solution for at least 1 min (Paragraph 5.5.1) before the analysis of each sample (see Note to Paragraph 7.3). Also, the analyst analyzes the instrument check standard (5.6) and the calibration blank (5.5.1) after each 10 samples.

7.6 Calculations: If dilutions were performed, the appropriate factors are applied to sample values. All results are reported in μg/L with up to three significant figures.

8.0 QUALITY CONTROL

8.1 All quality control data is maintained and available for easy reference or inspection.

8.2 Samples that are more concentrated than the linear calibration limit are diluted and reanalyzed unless the analyst uses an alternate, less sensitive line for which quality control data is already established.

8.3 The analyst employs a minimum of one laboratory blank per sample batch to determine if contamination or any memory effects are occurring.

8.4 One duplicate sample is analyzed for every 20 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

8.5 Whenever a new or unusual sample matrix is encountered, the analyst may perform a series of tests prior to reporting concentration data for analyte elements. These tests, as outlined in 8.5.1 through 8.5.3, will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.5.1 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

8.5.2 Matrix spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The use of a standard-addition analysis procedure can usually compensate for this effect.

CAUTION: The stand-addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

8.5.3 Standard addition: The standard-addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume V_x, are

taken. To the first (labeled A) is added a small volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculate:

$$c_x = \frac{S_B V_s C_s}{(S_A - S_B)\ V_x}$$

Where:

S_A and S_B = analytical signals (corrected for the blank) of solutions A and B, respectively.

V_S and c_S = should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

For the results of this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.

2. The chemical form of the analyte added must respond the same way as the analyte in the sample.

3. The interference effect must be constant over the working range of concern.

4. The signal must be corrected for any additive interference.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentration of the know standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown is Figure 1.

8.6 The instrument standardization is checked by analyzing appropriate quality control check standards as follows.

8.6.1 Instrument calibration is checked using a calibration blank and two appropriate standards.

8.6.2 Calibration is verified every 10 samples and at the end of the analytical run, using a calibration blank (5.5.1) and a single point check standard (5.6).

8.6.2.1 The results of the check standard are to agree within 10% of the expected value; if not, the analyst terminates the analysis, corrects the problem and recalibrates the instrument.

8.6.2.2 The results of the calibration blank are to agree within three standard deviations of the mean blank value. If not, the analyst repeats the analysis two more times and averages the results. If the average is not within three standard deviations of the background mean, the analyst terminates the analysis, corrects the problem, recalibrates, and reanalyzes the previous 10 samples.

8.6.3 The interelement and background correction factors are analyzed at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. The analyst does this by analyzing the interference check sample (Paragraph 5.7). Results should be within ± 20% of the true value obtained in 8.6.2.1.

8.6.4 Duplicate spiked samples are to be analyzed at a frequency of 205.

8.6.4.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate).

(A control limit of ± 20% for RPD shall be used for sample values greater than 10 times the instrument detection limit.)

8.6.4.2 The duplicate matrix spike sample recovery is to be within ± of the actual value.

8.6.5 The method of standard addition (Paragraph 8.5.3) is be used for the analysis of all EP extracts.

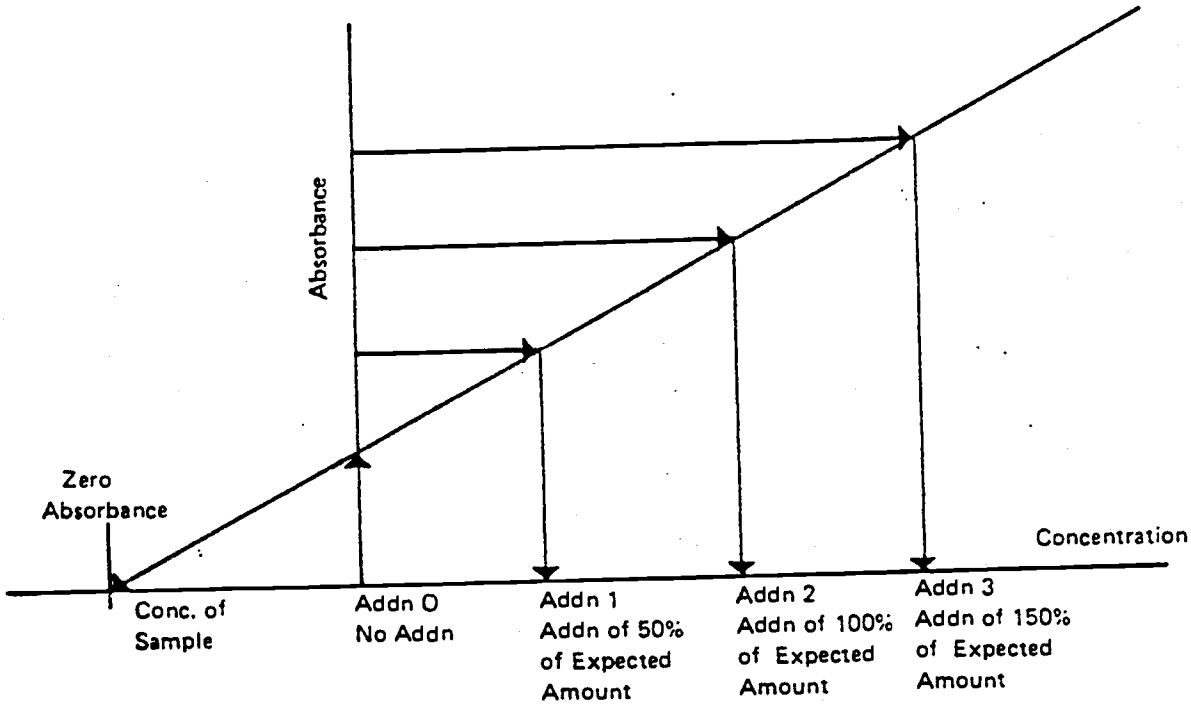


Figure 1. Standard Addition Plot.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 In a single laboratory evaluation, seven wastes were analyzed for 22 elements by this method. The mean percent relative standard deviation from triplicate analyses for all elements and wastes was 9 ± 2%. The mean percent recovery of spiked elements for all wastes was 93 ± 6%. Spike levels ranged from 100 µg/L to 100 mg/L. The wastes included sludges and industrial wastewaters.

Bibliography

1. Winge, R.K., V.J. Peterson, and V.A. Fassel, Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines, Final Report, March 1977-February 1978, Ames Laboratory, Ames, IA, sponsored by Environmental Research Laboratory, Athens, GA, EPA-600-4-78-017, March 1979.
2. Methods for Chemical analysis of Water and Wastes, EPA-600/4-82-05, December 1982, Method 200.7.
3. Patel, B.K., Raab, G.A., et al., Report on a Single Laboratory Evaluation of Inductively Coupled Optical Emission Method 6010, EPA Contract No. 68-03-3050, December 1984.
4. Chemron Inc. - Standard Operating Procedure (SOP) 846.

Date - December 6, 1994

Approved: _____ Date: 12/06/94

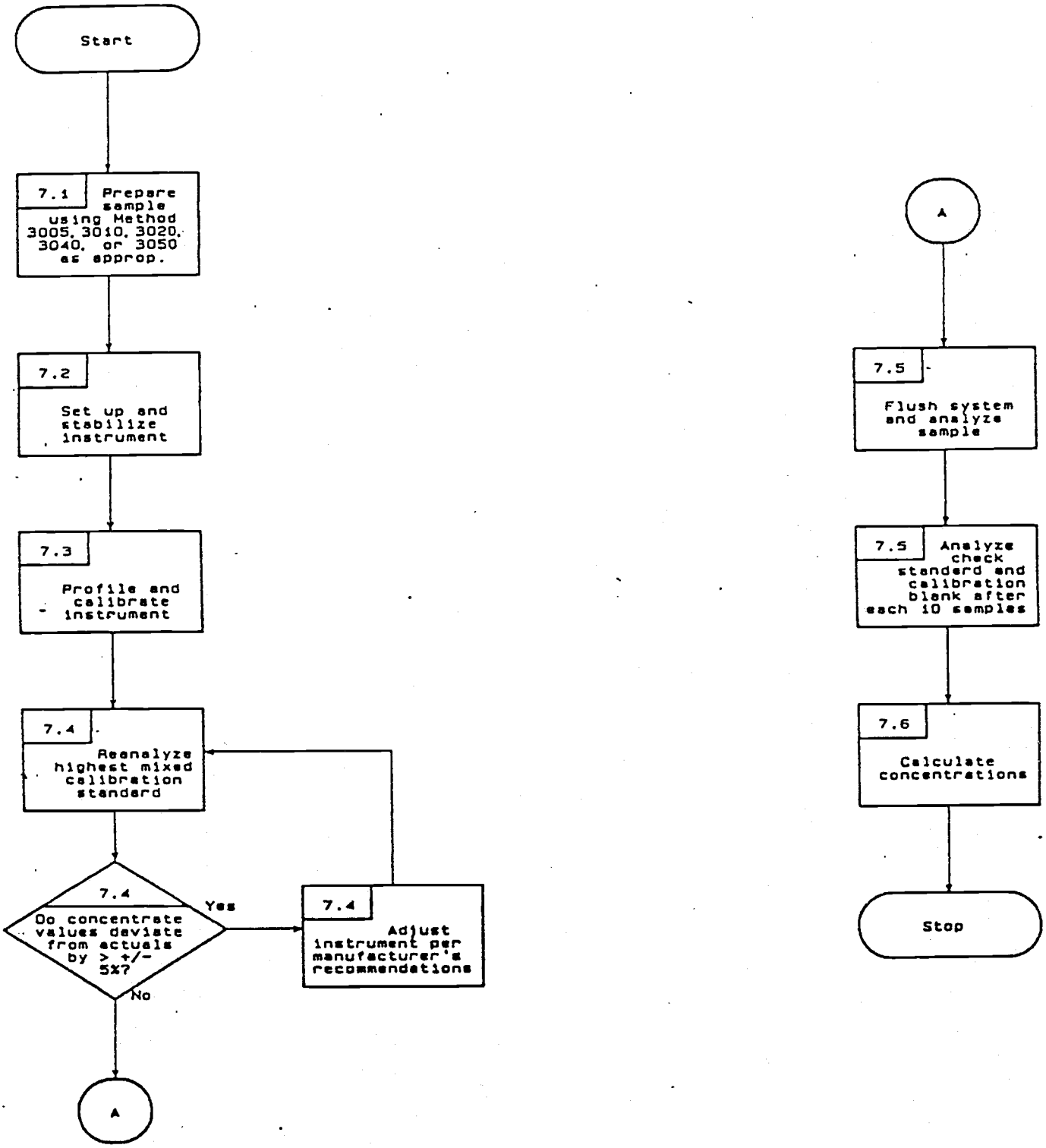
TABLE 4. ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1 True Value (ug/L)	Sample No. 1 Mean Reported Value (ug/L)	Sample No. 1 Mean SD[b] (%)	Sample No. 2 True Value (ug/L)	Sample No. 2 Mean Reported Value (ug/L)	Sample No. 2 Mean SD[b] (%)	Sample No. 3 True Value (ug/L)	Sample No. 3 Mean Reported Value (ug/L)	Sample No. 3 Mean SD[b] (%)
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	700	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se[c]	40	32	21.9	6	8.5	42	10	8.5	8.3

[a]Not all elements were analyzed by all laboratories.

[b]SD = standard deviation.

[c]Results for Se are from two laboratories.


Start
7.1 Prepare sample using Method 3005, 3010, 3020, 3040, or 3050 as approp.
7.2 Set up and stabilize instrument
7.3 Profile and calibrate instrument
7.4 Reanalyze highest mixed calibration standard
7.4 Do concentrate values deviate from actuals by > +/- 5%?
Yes
No
7.4 Adjust instrument per manufacturer's recommendations
A
A
7.5 Flush system and analyze sample
7.5 Analyze check standard and calibration blank after each 10 samples
7.6 Calculate concentrations
Stop

CHEMRON INC. - STANDARD OPERATING PROCEDURE 7470

MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (SOP 7470 can also be used for analyzing certain solid and sludge-type wastes; however, SOP 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

2.2 SOP 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

3.2 Copper has also been reported to interfere; however, copper concentration as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrometer: Any atomic absorption unit with an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable-speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of he cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-w bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10°C above ambient.

4.8 The cold-vapor generator is assembled as shown in Figure 1.

4.8.1 The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system.

4.8.2 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor into an exhaust hood or to pass the vapor through some absorbing medium, such as:

1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4; or
2. 0.25% Iodine in a 3% KI solution.

A specially treated charcoal that will absorb mercury vapor is also available from Barnebey and Cheney, East 8th Avenue and North Cassidy Street, Columbus, Ohio 43219, Cat. #580-22.

5.0 REAGENTS

5.1 ASTM Type II water (D1193): Water should be monitored for impurities.

5.2 Sulfuric acid (H_2SO_4), concentrated: Reagent grade.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.

5.4 Nicric acid (HNO_3), concentrated: Reagent grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

5.5 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N H_2SO_4. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

5.6 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in Type II water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

5.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of Type II water.

5.8 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of Type II water.

5.9 Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of Type II water. Add 10 mL of concentrated HNO_3 and adjust the volume to 100.0 mL (1 mL = 1 mg Hg).

5.10 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 g per mL. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask, as needed, before addition of the aliquot.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample holding times, digestion requirements and preservatives are summarized below.

Metal Measurement	Digestion, mL	Preservative	Holding Time
Total Recoverable	100	Nitric pH <2	6 mo.
Dissolved	100	Filter on Site Nitric pH <2	6 mo.
Suspended	100	Filter on Site	6 mo.

Solid samples require no preservation other than storing at 4^0C until analyzed.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of <2 with HNO_3. The suggested maximum holding times for these samples are 38 days in glass containers and 13 days in plastic containers.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Sample preparation: Transfer 100 mL, or an aliquot diluted to 100 mL, containing <1.0 g of mercury, to a 300-mL BOD bottle. Add 5 mL of H_2SO_4 and 2.5 mL of concentrated HNO_3, mixing after each addition. Add 15 mL of potassium permanganate solution to each sample bottle. Sewage samples may require additional permanganate. Ensure that equal amounts of permanganate are added to standards and blanks. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. After a delay of at least 30 sec, add 5 mL of stannous sulfate, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.2 Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-mL aliquots of the mercury working standard, containing 0-1.0 μg of mercury, to a series of 300-mL BOD bottles. Add enough Type II water to each bottle to make a total volume of 100 mL. Mix thoroughly and add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to each bottle. Add 15 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized, wait 30 sec, add 5 mL of the stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.3 Analysis: At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum valve. Close the bypass valve, remove the stopper and frit from the BOD bottle, and continue the aeration.

7.4 Construct a calibration curve by plotting the absorbances of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

7.5 Analyze all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences by the method of standard additions.

7.6 duplicates, spiked samples, and check standards should be routinely analyzed.

7.7 Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 μg/g dry weight).

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process.

8.7 Method of standard additions:

8.7.1 In the simplest version of this method, equal volumes of sample are added to a deionized distilled (Type II) water blank and to a standard (refer to Paragraph 8.7.3). If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the

concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown if Figure 1.

8.7.2 The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

a. The absorbance plot of sample and standards must be linear over the concentration range of concern For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

b. The effect of the interference should not vary as the ration of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

c. The determinations must be free of spectral interference and corrected for nonspecific background interference.

8.7.3 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B)\ V_x}$$

Where:

S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively.

V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix.

If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in SOP 245.1 of Methods for Chemical Analysis of Water and Wastes.

Bibliography

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 245.1.
2. Chemron Inc. Standard Operating Procedure 7471.
3. Chemron Inc. Standard Operating Procedure 245.1.

Revision 2.0

Date - December 7, 1994

Approved: _____ Date: 12/07/94

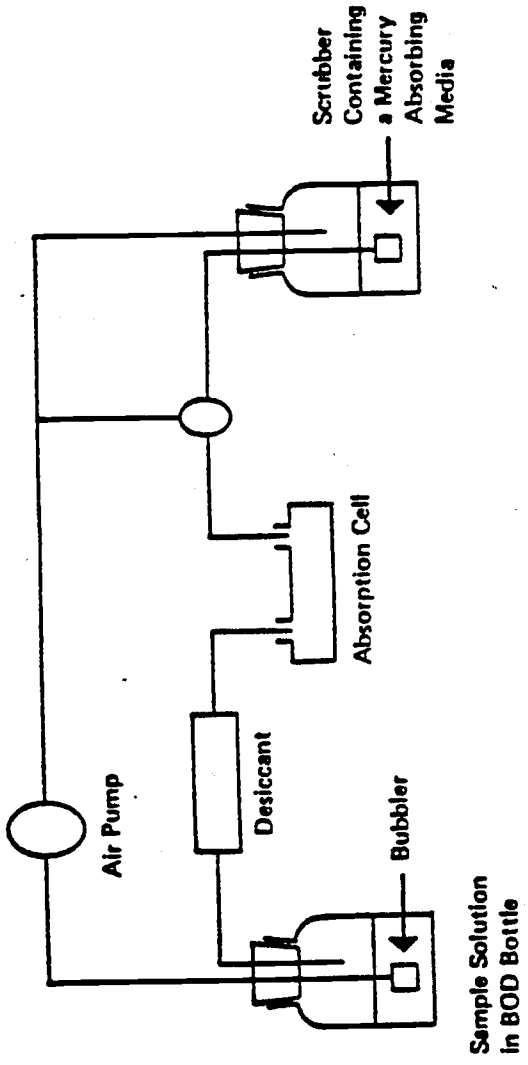
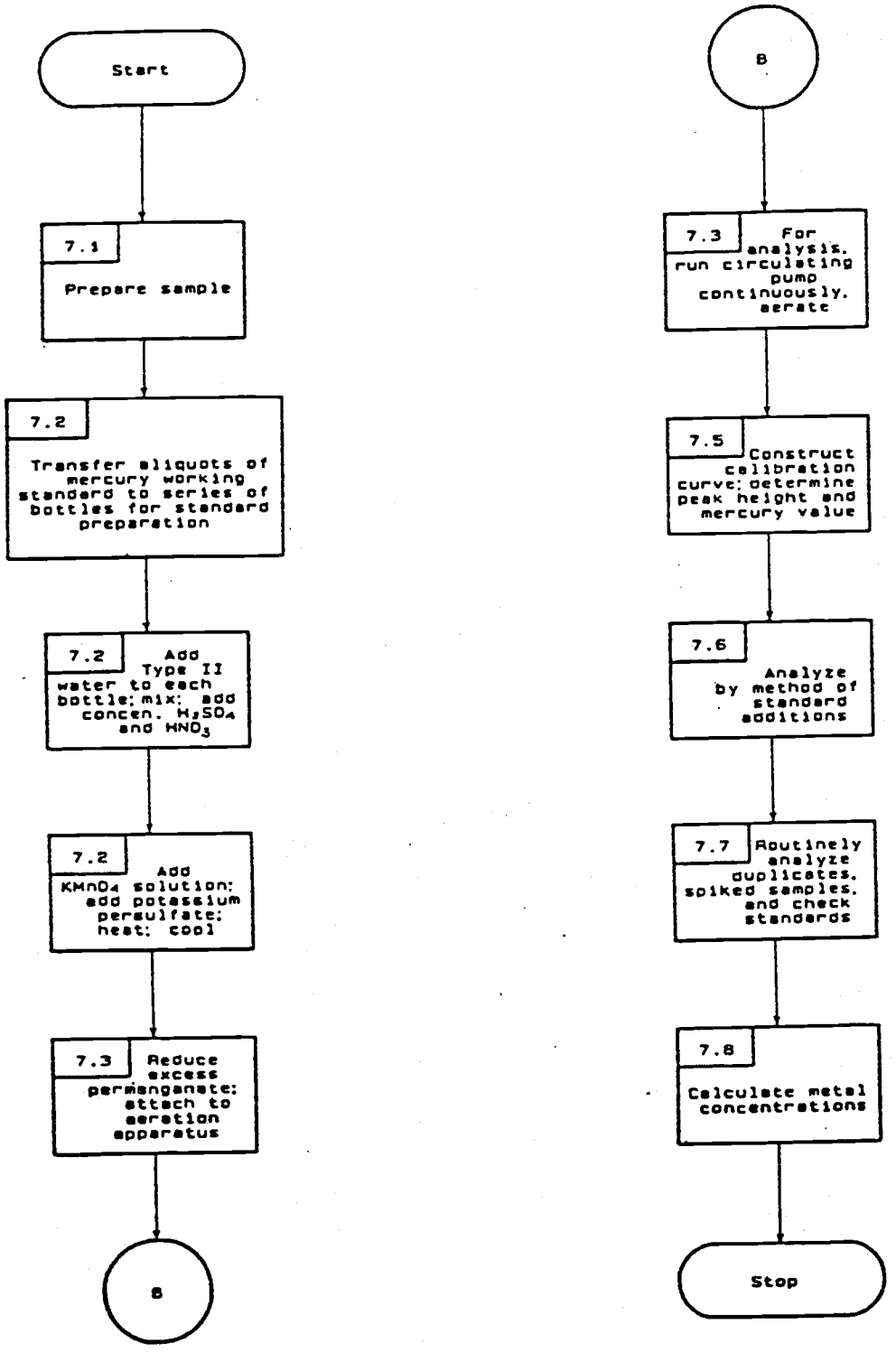


Figure 1. Apparatus for flameless mercury determination.


Start
7.1 Prepare sample
7.2 Transfer aliquots of mercury working standard to series of bottles for standard preparation
7.2 Add Type II water to each bottle; mix; add concen. H_2SO_4 and HNO_3
7.2 Add $KMnO_4$ solution; add potassium persulfate; heat; cool
7.3 Reduce excess permanganate; attach to aeration apparatus
A
B
7.3 For analysis, run circulating pump continuously, aerate
7.5 Construct calibration curve; determine peak height and mercury value
7.6 Analyze by method of standard additions
7.7 Routinely analyze duplicates, spiked samples, and check standards
7.8 Calculate metal concentrations
Stop

CHEMRON INC. - STANDARD OPERATING PROCEDURE 7471

MERCURY IN SOLID OR SEMISOLID WASTE
(MANUAL COLD-VAPOR TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the solid or semisolid samples must be prepared according to the procedure discussed in this method.

2.2 SOP 7471, a cold-vapor atomic absorption method, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high s 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

3.2 Copper has also been reported to interfere; however, copper concentration as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrometer: Any atomic absorption unit with an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable-speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of he cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-w bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10°C above ambient.

4.8 The cold-vapor generator is assembled as shown in Figure 1.

4.8.1 The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system.

4.8.2 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor into an exhaust hood or to pass the vapor through some absorbing medium, such as:

1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4; or
2. 0.25% Iodine in a 3% KI solution.

A specially treated charcoal that will absorb mercury vapor is also available from Barnebey and Cheney, East 8th Avenue and North Cassidy Street, Columbus, Ohio 43219, Cat. #580-22.

5.0 REAGENTS

5.1 ASTM Type II water (D1193): Water should be monitored for impurities.

5.2 Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.

5.4 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is in a suspension and should be stirred continuously during use. A 10% solution of stannous chloride can be substituted for stannous sulfate.)

5.5 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in Type II water and dilute to 100 mL. Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

5.6 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of Type II water.

5.7 Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of Type II water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL (1 mL = 1 mg Hg).

5.10 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg/per mL. This working standard and the dilution of the stock mercury solutions should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask, as needed, before adding the aliquot.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample holding times, digestion requirements and preservatives are summarized below.

Metal Measurement	Digestion, mL	Preservative	Holding Time
Total Recoverable	100	Nitric pH <2	6 mo.
Dissolved	100	Filter on Site Nitric pH <2	6 mo.
Suspended	100	Filter on Site	6 mo.

Solid samples require no preservation other than storing at 4°C until analyzed.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of <2 with nitric acid.

6.4 For solids or semisolids, moisture may be driven off in a drying oven at a temperature of 60°C.

7.0 PROCEDURE

7.1 Sample preparation: Weigh triplicate 0.2-g portions of untreated sample and place in the bottom of a BOD bottle. Add 5 mL of Type II water and 5 mL of aqua regia. Heat 2 min in a water bath at 95°C. Cool; then and add 50 mL of Type II water and 15 mL potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 min at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate.

CAUTION: Do this addition under a hood, as Cl_2 could be evolved. Add 55 mL of Type II water. Treating each bottle individually, add 5 mL of stannous sulfate and immediately attach the bottle to the aeration apparatus. Continue as described under step 7.4.

7.2 An alternate digestion procedure employing an autoclave may also be used. In this method, 5 mL of concentrated H_2SO_4 and 2 mL of concentrated HNO_3 are added to the 0.2 g of sample. Add 5 mL of saturated $KMnO_4$ solution and cover the bottle wit a piece of aluminum foil. The samples are autoclaved at 121°C and 15 lb for 15 min. Cool, dilute to a volume of 100 mL with Type II water, and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Purge the dead air space and continue as described under step 7.4.

7.3 Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-mL aliquots of the mercury working standard, containing 0-1.0 ug of mercury, to a series of 300-mL BOD bottles. Add enough Type II water to each bottle to make a total volume of 10 mL. Add 5 mL of aqua regia and heat 2 min in a water bath at 95°C. Allow the sample to cool; add 50 mL Type II water and 15 mL of $KMnO_4$ solution to each bottle and return to the water bath for 30 min. cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Add 50 mL of Type II water. Treating each bottle individually, add 5 mL of stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as describe in Step 7.4.

7.4 Analysis: At this point, the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance, as exhibited either on the spectrophotometer or the recorder, will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the

aeration until the absorbance returns to its minimum valve. Close the bypass valve, remove the fritted tubing from the BOD bottle, and continue the aeration.

7.5 Construct a calibration curve by plotting the absorbances of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

7.6 Analyze all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences by the method of standard additions.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 μg/g dry weight).

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process.

8.7 Method of standard additions:

8.7.1 In the simplest version of this method, equal volumes of sample are added to a deionized distilled (Type II) water blank and to a standard (refer to Paragraph 8.7.3). If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown if Figure 1.

8.7.2 The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

a. The absorbance plot of sample and standards must be linear over the concentration range of concern For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

b. The effect of the interference should not vary as the ration of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

c. The determinations must be free of spectral interference and corrected for nonspecific background interference.

8.7.3 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B)\ V_x}$$

Where:

S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively.

V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix.

If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 245.1 of Methods for Chemical Analysis of Water and Wastes.

9.2 The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended so show the precision of the combined sample preparation and analysis method.

Bibliography

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 245.5.
2. Gaskill, A ., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

Revision 2.0

Date - December 7, 1994

Approved: _____ Date: 12/07/94

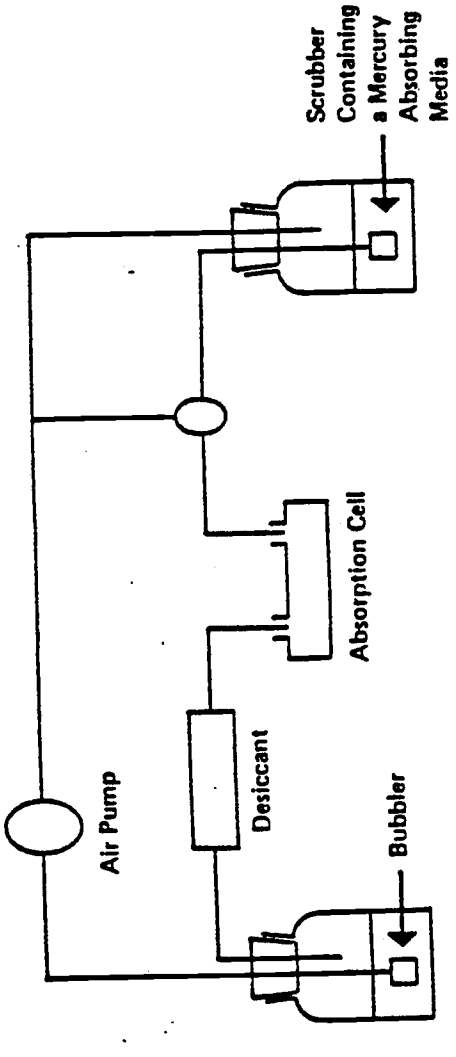
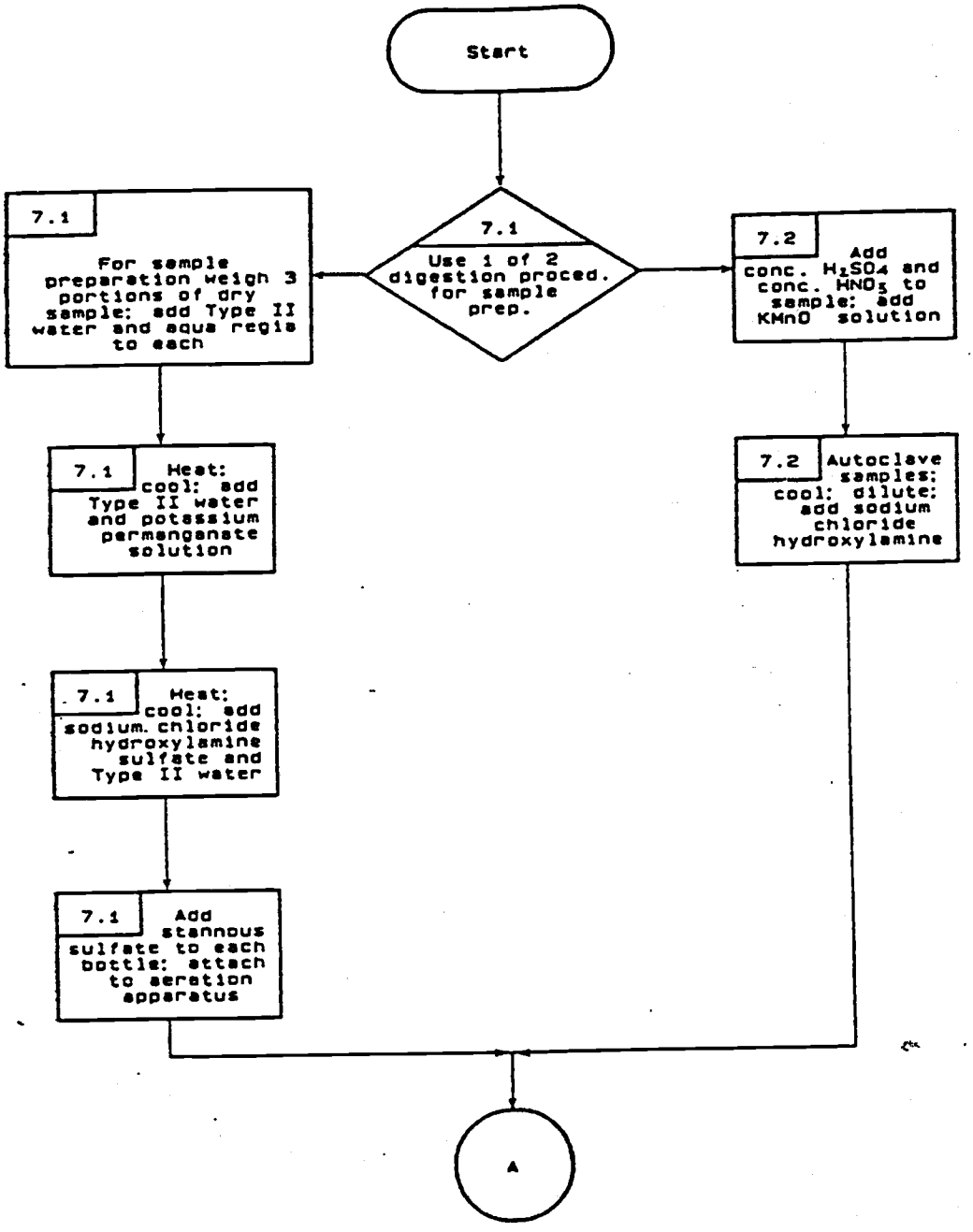
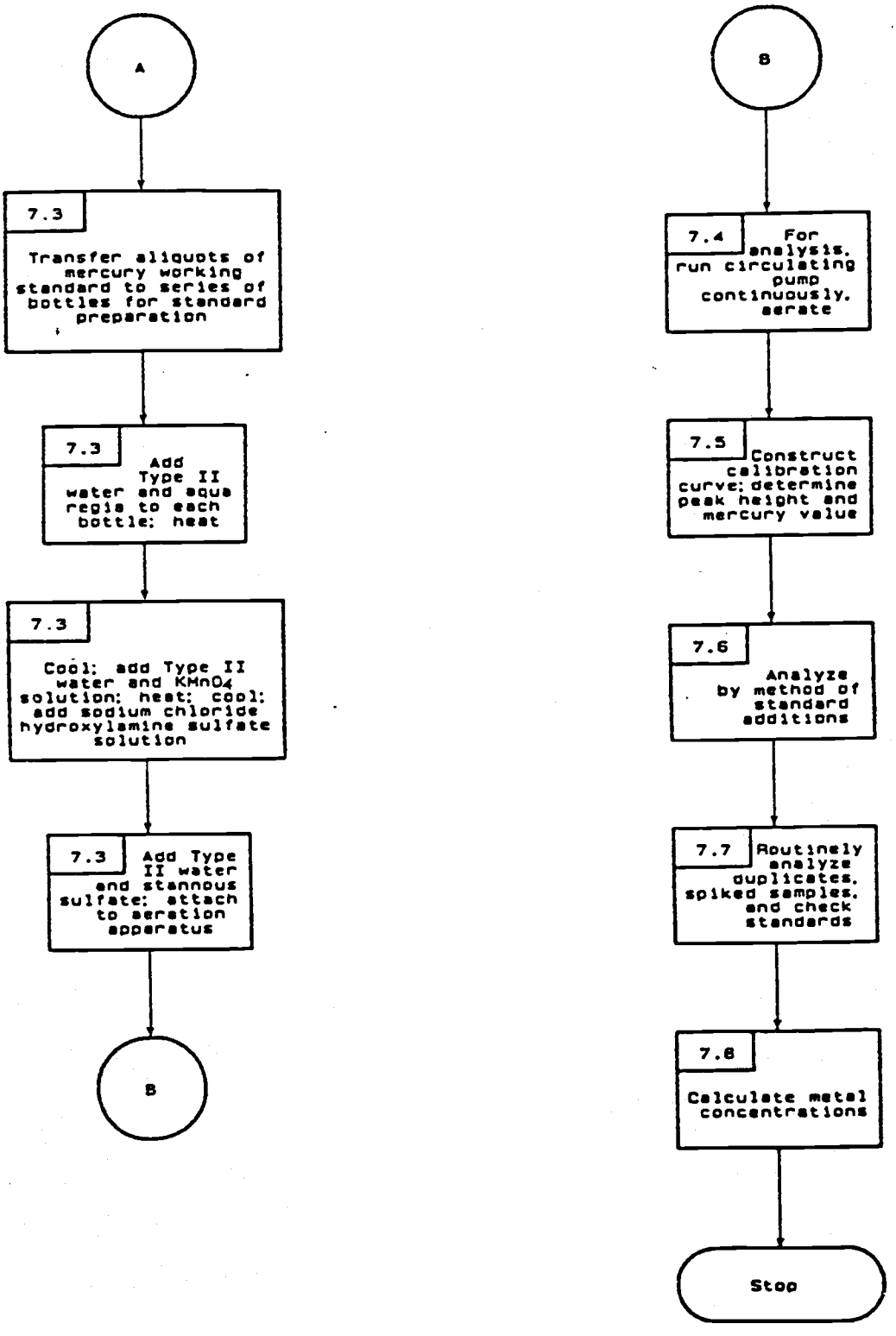


Figure 1. Apparatus for flameless mercury determination.

Sample Matrix	Preparation Method	Laboratory Replicates
Emission control dust	Not known	12, 12 ug/g
Wastewater treatment sludge	Not known	0.4, 0.28 ug/g


Start
7.1
Use 1 of 2 digestion proced. for sample prep.
7.1
For sample preparation weigh 3 portions of dry sample: add Type II water and aqua regia to each
7.2
Add conc. H_2SO_4 and conc. HNO_3 to sample: add $KMnO_4$ solution
7.1
Heat: cool: add Type II water and potassium permanganate solution
7.2
Autoclave samples: cool: dilute: add sodium chloride hydroxylamine
7.1
Heat: cool: add sodium chloride hydroxylamine sulfate and Type II water
7.1
Add stannous sulfate to each bottle: attach to aeration apparatus
A


A
7.3
Transfer aliquots of mercury working standard to series of bottles for standard preparation
7.3
Add Type II water and aqua regia to each bottle: heat
7.3
Cool: add Type II water and KMnO4 solution: heat: cool: add sodium chloride hydroxylamine sulfate solution
7.3
Add Type II water and stannous sulfate: attach to aeration apparatus
B
B
7.4
For analysis, run circulating pump continuously, aerate
7.5
Construct calibration curve: determine peak height and mercury value
7.6
Analyze by method of standard additions
7.7
Routinely analyze duplicates, spiked samples, and check standards
7.8
Calculate metal concentrations
Stop

CHEMRON INC. - STANDARD OPERATING PROCEDURE 8080

ORGANOCHLORINE PESTICIDES AND PCB's

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). Table 1 indicates compounds that may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 SOP 8080 provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides and PCBs. Prior to the use of SOP 8080, appropriate sample extraction techniques are used. Both neat and diluted organic liquids (SOP 3580, Waste Dilution) may be analyzed by direct injection. A 2-μL sample is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of SOP 8080 usually depends on the level of interferences rather than on instrumental limitation. If interferences prevent detection of the analytes, SOP 8080 may also be performed on samples that have undergone cleanup. Chemron uses SOP 3620, Florisil Column Cleanup, to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Refer to SOPs 3500 (Section 3.5, in particular), 3600, and Method 8000 in the SW-846 EPA Compendium for more information.

3.2 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks, especially in the 15% and 50% fractions from the Florisil cleanup. Common flexible plastics contain varying mounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences

from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. The contamination from phthalate esters can be completely eliminated with a microcoulometric or electrolytic conductivity detector.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas chromatograph: Chemron is equipped with an analytical system complete with the Hewlett-Packard Model 5890 Series II Gas Chromatograph, a GC suitable for splitless injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. Chemron utilizes an HP Integrator Model 3396 Series II for measuring peak areas.

4.1.2 Columns:

4.1.2.1 Column 1: DB-608 (J&W PN 123-1730) 0.32 mmidx. 30 m X 0.5 um.

4.1.2.2 Column 2: DB-5ms (J&W PN 128-5522) 0.20 mmidx. 25 m X 0.33 um.

4.1.3 Detectors: Electron capture detector (ECD).

4.2 Kuderna-Danish (K-D) apparatus:

4.2.1 Concentrator tube: 10-mL, graduated (Kontes K-570050-1025). Ground-glass stopper is used to prevent evaporation of extracts.

4.2.2 Evaporation flask: 500-mL (Kontes K-570001-500). Attach to concentrator tube with springs.

4.2.3 Snyder column: Three-ball macro (Kontes K-503000-0121).

4.2.4 Snyder column: Two-ball micro (Kontes K-569001-0219).

4.3 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide).

4.4 Water bath: The bath is used in a hood.

4.5 Volumetric flask: 10-, 50-, and 100-mL with a ground-glass stopper.

4.6 Microsyringe: 10-μL.

4.7 Syringe: 5-mL.

4.8 Vials: Glass, 2-, 10-, and 20-mL capacity with Teflon-lined screw cap.

5.0 REAGENTS

5.1 Solvents: Hexane, acetone, toluene, isoctane (2,2,4-trimethylpentane) (pesticide quality).

5.2 Stock standard solutions:

5.2.1 The analyst uses purchased certified standard solutions. For single component pesticides: Restek Cat No. 31012-SV, 2.0 mg/mL. For multi-component pesticides and PCB's: Toxaphene - Ultra Scientific Cat No. EPA-1161, 1.0 mg/mL; Chlordane - Ultra Scientific Cat No. EPA-1086, 5.0 mg/mL; Aroclors as listed below:

Aroclor No.	Supplier	Cat. No.	Conc. (mg/mL)
1016	Restek Corp.	32006	1.0
1221	Restek Corp.	32007	1.0
1232	Restek Corp.	32008	1.0
1242	Restek Corp.	32009	1.0
1248	Restek Corp.	32010	1.0
1254	Restek Corp.	32011	1.0
1260	Restek Corp.	32012	1.0

5.2.2 The stock standard solutions are transfered into Teflon-lined screw-cap bottles. They are then stored at 4°C and protected from light. Stock standards are checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.2.3 Stock standard solutions are replaced after one year, or sooner if comparison with check standards indicates a problem.

5.3 Calibration standards: Calibration standards of five concentration levels for each parameter of interest are prepared through dilution of the stock standards with acetone. One of the concentration levels is at a concentration near, but above, the method detection limit. The remaining concentration levels correspond to the expected range of concentrations found in the real samples or define the working range of the GC. Calibration solutions are replaced after six months, or sooner, if comparison with check standards indicates a problem.

5.5 Surrogate standards: The analyst monitors the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with pesticide surrogates. Because GC/ECD data are much more subject to interference than GC/MS, a secondary surrogate is to be used when sample interference is apparent. Dibutyl-chlorendate (DBC) is also subject to acid and base degradation. Therefore, two surrogate standards are added to each sample; however, only one need be calculated for recovery. DBC is the primary surrogate and is used whenever possible. However, 2,4,5,6-tetrachloro-meta-xylene is also evaluated for acceptance. If both surrogates are out of limits for a sample, the analyst takes the appropriate corrective action (Section 8.3). SOP 3500, Section 5.3.2, indicates the proper procedure for preparing these surrogates.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the chapter, Organic Analytes, Section 4.1 in the SW-846 EPA Compendium. Extracts are stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two in the SW-846 EPA Compendium for information on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral, or as is, pH with methylene chloride, using either SOP 3510 or 3520. Solid samples are extracted using either SOP 3540 or 3550.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent is exchanged to hexane. The exchange is performed during the K-D procedures listed in all of the extraction methods or automatically with the Zymark Turbovap Concentrators. The exchange with KD is performed as follows.

7.1.2.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, the analyst allows the apparatus to cool and drain for at least 10 min.

7.1.2.2 The temperature of the hot water bath is increased to about 90°C. The analyst momentarily removes the Snyder column, adds 50 mL of hexane, a new boiling chip, and reattaches the macro-Snyder column. The extract is concentrated using 1 mL of hexane to prewet the Snyder column. The analyst then places the K-D apparatus on the water bath so that the vertical position of the apparatus and the water temperature, as required, will complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, the analyst removes the K-D apparatus and allow it to drain and cool for at least 10 min.

7.1.2.3 Next, the analyst removes the Snyder column and rinses the flask and its lower joint into the concentrator tube with 1-2 mL of hexane. A 5-mL syringe is recommended for this operation. The analyst then adjusts the extract volume to 10.0 mL. The analyst stoppers the concentrator tube and stores refrigerated at 4°C, if further processing will not be performed immediately. If the extract will be stored longer than two days, it is transferred to a Teflon-sealed screw-cap vial. The analyst then proceeds with gas chromatographic analysis if further cleanup is not required.

7.2 Gas chromatography conditions:

7.2.1 Column 1:

Carrier gas (UHP Nitrogen) flow rate: 1.0 mL/min at 8 psig.

Column temperature program:

Initial Temp	= 150°C for 1 minute
Ramp Rate 1	= 5°/min to 250°C for 2 minutes
Ramp Rate 2	= 2.5°/min to 280°C for 5 minutes

Make-up: 34 mL/min

7.2.2 Column 2:

Carrier gas (UHP Nitrogen) flow rate: .35 mL/min

Column temperature program:

Initial Temp	= 150°C for 1 minute
Ramp Rate 1	= 10°/min to 200°C for 2 minutes
Ramp Rate 2	= 2.5°/min to 250°C
Ramp Rate 3	= 25°/min to 300°C for 5 minutes

7.3 Calibration: Refer to SOP 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 Chemron uses the external calibration procedure. Method 8000 provides a description of this procedures.

7.4 Gas chromatographic analysis:

7.4.1 SOP 8000 (section 7.6) provides an explanation of the analysis sequence, appropriate dilutions, establishment of daily retention time windows,and identification criteria. The analyst includes a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.2 Examples of GC/ECD chromatograms for various pesticides and PCBs are shown in Figures 1 through 5.

7.4.3 DDT and endrin are easily degraded in the injection port if the injection prot or front of the column is dirty. This is the result of buildup of high boiling residue from sample injection. The analyst checks for degradation problems by injecting 2 uL of 1:100 diluted performance evaluation mix (Ultra-Scientific Cat No. CLP 231-1) 4,4'-DDT and endrin, looking for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). If degradation of either DDT or endrin exceeds 20%, the analyst takes corrective action before proceeding with calibration, by following the GC system maintenance outlined in Section 7.7 of SOP 8000. Percent breakdown is calculated as follows:

$$\%\ breakdown\ for\ 4,4'\text{-}DDT = \frac{Total\ DDT\ degradation\ peak\ area\ (DDE + DDD)}{Total\ DDT\ peak\ area\ (DDT + DDE\ DDD)} \times 100$$

$$\%\ breakdown\ for\ Endrin = \frac{Total\ endrin\ degradation\ peak\ area\ (endrin\ aldehyde + endrin\ keytone)}{Total\ endrin\ peak\ area\ (endrin + endrin\ aldehyde + endrin\ keytone)} \times 100$$

7.4.4 The sample volume injected and the resulting peak sizes are (in area units or peak heights) are recorded.

7.4.5 Using the external calibration procedure (SOP 8000), the analyst determines the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

7.4.6 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo cleanup using SOP 3620. The resultant extract(s) may be analyzed by GC directly or may undergo further cleanup to remove Sulfur using SOP 3660.

7.5 Cleanup:

7.5.1 The analyst proceeds with SOP 3620, followed by, if necessary, SOP 3660, using the 10-mL hexane extracts obtained from Paragraph 7.1.2.3.

7.5.2 Following cleanup, the extracts are analyzed by GC, as described in the previous paragraphs and in SOP 8000.

7.5.3 If only PCB's are to be measured in a sample, Florisil Cleanup (SOP 3620), is performed.

7.6 Calculations (exerpted from U.S. FDA, PAM):

7.6.1 Calculation of Certain Residues: Residues which are mixtures of two or more components present problems in measurement. When they are found together, e.g., toxaphene and DDT, the problem of quantitation becomes even more difficult. In the following sections suggestions are offered for handling toxaphene, chlordane, PCB, DDT, and BHC. A column 10% DC-200 stationary phase was used to obtain the chromatograms in Figures 6-9.

7.6.2 Toxaphene: Quantitative calculation of toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene on GC/ECD: (a) adjust sample size so that toxaphene major peaks are 10-30% full-scale deflection (FSD); (b) inject a toxaphene standard that is estimated to be within ± 10 ng of the sample; (c) construct the baseline of standard toxaphene between it extremities; and (d) construct the baseline under the sample, using the distances of the peaks troughs to baseline on the standard as a guide (Figures 7, 8, and 9). this procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard. A toxaphene standard that has been passed through a Florisil column will show a shorter retention time for peak X and an enlargement of peak Y.

7.6.3 Toxaphene and DDT: If DDT is present, it will superimpose itself on toxaphene peak V. To determine the approximate baseline of the DDT, draw a line connecting the trough of peaks U and V with the trough of peaks W and X and construct another line parallel to this line which will just cut the top of peak W (Figure 61). this procedure was tested with ratios of standard toxaphene-DDT and toxaphene by the "parallel lines" method of baseline construction were within 10% of the actual values in all cases.

7.6.3.1 A series of toxaphene residues have been calculated using total peak area for comparison to the standard and also using area of the last four peaks only in both sample and standard. The agreement between the results obtained by the two methods justifies the use of the latter method for calculating toxaphene in a sample where the early eluting portion of the toxaphene chromatogram is interfered with by other substances.

7.6.3.2 The baseline for methoxychlor superimposed on toxaphene (Figure 8b) was constructed by overlaying the samples on a toxaphene standard of approximately the same concentration (Figure 8A) and viewing the charts against a lightened background.

7.6.4 Chlordane is a technical mixture of at least 11 major components and 30 or more minor ones. Gas chromatography-mass spectrometry and nuclear magnetic resonance analytical techniques have been applied to the elucidation of the chemical structures of the many chlordane constituents. Figure 9a is a chromatogram of standard chlordane. Peaks E and F are responses to trans- and cis-chlordane, respectively. These are the two major component of technical chlordane, but the exact percentage of each in the technical material is not completely defined and is not consistent from batch to batch. Other labelled peaks in Figure 9a are thought to represent: A, monochlorinated adduct of pentachlorocyclopentadiene with cyclopentadiene; B, coelution of heptachlor and a-chlordane; C, coelution of p-chlordane and y-chlordane; D, a chlordane analog; G, coelution of cis-nonachlor and "Compound K," a chlordane isomer. The right "shoulder" of peak F is caused by transnonachlor.

7.6.4.1 The GC pattern of a chlordane residue may differ considerable from that of the technical standard. Depending on the sample substrate and its history, residues of chlordane can consists of almost any combination of: constituents form the technical chlordane; plant and/or animal metabolites; and products of degradation caused by exposure to environmental factors such as water and sunlight. Only limited information is available on which residue GC patterns are likely to occur in which samples types, and

even this information may not be applicable to a situation where the route of exposure is unusual. For example, fish exposed to a recent spill of technical chlordane will contain residue drastically different from a fish whose chlordane residue was accumulated by ingestion of small fish or of vegetation, which in turn had accumulated residues because chlordane was in the water from agricultural runoff.

7.6.4.2 Because of this inability to predict a chlordane residue GC pattern, it is not possible to prescribe a single method residue GC pattern, it is not possible to prescribe a single method for the quantitation of chlordane residues. The analyst must judge whether or not the residue's GC pattern is sufficiently similar to that of a technical chlordane reference material tho use the latter as a reference standard for quantitation.

7.6.4.3 When the chlordane residue does <u>not</u> resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, quantitate each peak separately against the appropriate reference materials and report the individual residues. (Reference materials are available for at least 11 chlordane constituents, metabolites or degradation products which may occur in the residue.)

7.6.4.4 When the GC pattern of the residue resembles that of technical chlordane, quantitate chlordane residues by comparing the total area of the chlordane chromatogram from peaks A through F (Figure 9a) in the sample versus the same part of the standard chromatogram. Peak G may be obscured in a sample by the presence of other pesticides. If G is not obscured, include it in the measurement for both standard and sample. If the heptachlor epoxide peak is relatively small, include it as part of the total chlordane area for calculation of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion as in Figure 6j, calculate theses separately and subtract their areas from total area to give a corrected chlordane area. (Note that octachlor epoxide, metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.)

7.6.4.5 To measure the total area of the chlordane chromatogram, proceed as in Section 7.6.2 on toxaphene. Inject an amount of technical chlordane standard which will produce a chromatogram in which peaks E and F are approximately the same size as those in the sample chromatograms. construct the baseline beneath the standard from the beginning of peak A to the end of peak F as shown in Figure 9a. Use the distance from the trough between peaks E and F to the baseline in the chromatogram of the standard to construct the baseline in the chromatogram of the

sample. Figure 9b shows how the presence of toxaphene causes the base line under chlordane to take an upward angel. When the size of peaks E and F in standard and sample chromatograms are the same, the distance from the through of the peaks to the baseline should be the same.Measurement of chlordane area should be one by total peak area if possible.

NOTE: A comparison has been made of the total peak area integration method and the addition of peak heights method for several samples containing chlordane. The peak heights A, B, C, D, E, and F were measured in millimeters from peak maximum of each to the baseline constructed under the total chlordane area and were then added together. These results obtained by the two techniques are to close to ignore this method of "peak height addition" as a means of calculating chlordane. The technique has inherent difficulties because not all the peaks are symmetrical and not all are present in the same ration in standard and in sample. this method does offer a means of calculating results if no means of measuring total area is practical.

7.6.5 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, Strobane, and chlordane: in each case, the chemical is made up of numerous compounds and so the chromatograms are multi-peak; also in each case the chromatogram of the residue may not match that of the standard.

7.6.5.1 Mixtures of PCB of various chlorine contents were sold for many years i the U.S. b the Monsanto Co. under the tradename Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometime found as residues in foods, especially fish.

7.6.5.2 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue. A choice must be made as to which Aroclor of mixture of Aroclors will produce a chromatogram most similar to that of the residue. This may also involve a judgement about what proportion of the different Aroclors to combine to produce the appropriate reference material.

7.6.5.3 Quantitate PCB residues by comparing total area of residue peaks to total area of peaks from appropriate Aroclor(s) reference materials. Measure total area or height response from common baseline under all peaks. Use only those peaks from sample that can be attributed to chlorobiphenyls. These peaks must also be present in chromatogram of reference materials. Mixture of Aroclors may be required to provide best match of GC patterns of sample and reference.

7.6.6 DDT: DDT found in samples often consists of both o,p'- and o,p'-DDT. Residues of DDE and TDE are also frequently present. Each isomer of DDT and its metabolites should be quantitated using the pure standard of that compound and reported as such.

7.6.7 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor; it consists of a mixture of six chemically distinct isomers and one or more heptachlor-cyclohexanes and octachloro-cyclohexanes.

7.6.7.1 Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. The elimination rate of the isomers fed to rats was 3 weeks for the a-,y-, and o- isomers and 14 weeks for the p-isomer. Thus it may be possible to have any combination of the various isomers in different food commodities. BHC found in dairy products usually has a large percentage of p-isomer.

7.6.7.2 Individual isomers (a, p, y, and o) were injected into has chromatographs equipped with flame ionization, microcoulometric, and electron capture detectors. Response for the four isomers is very nearly the same whether flame ionization or microcoulometric GLC is used. The a-, y-, and o-isomers show equal electron affinity. p-BHC shows a much weaker electron affinity compared to the other isomers.

7.6.7.3 Quantitate each isomer (a, p, y, and 0) separately against a standard of the respective pure isomer, using a GC column which separates all the isomers from one another.

7.6.8 Calibration Factors for Multi-Component Pesticides and PCB's

7.6.8.1 A set of 5 major peaks is selected for each multi-component analyte. Retention time and calibration factors are determined from the initial calibration analysis for each peak.

7.6.8.2 The chromatograms of the standards for the multi-component analytes analyzed during the initial calibration sequence must display most of the peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale.

8.0 QUALITY CONTROL

8.1 Chapter One of the SW-846 EPA Compendium describes specific quality control procedures. Quality control to validate sample extraction is covered in SOP 3500 and in the extraction method utilized. If extract cleanup is performed, the QC in SOP 3600 and in the specific cleanup method is followed.

8.2 Chemron performs the mandatory quality control to evaluate the GC system operation found in SOP 8000, Section 8.6.

8.2.1 The quality control check sample concentrate (SOP 8000, Section 8.6) contains each single-component parameter of interest at the following concentrations in acetone: 4,4'-DDD, 10 µg/mL; 4,4'-DDT, 10 µg/mL; endosulfan II, 10 µg/mL; endosulfan sulfate, 10 µg/mL; endrin, 10 µg/mL; and any other single-component pesticide, 2 µg/mL. If this SOP is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate will contain the most representative multi-component parameter at a concentration of 50 µg/mL in acetone.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables are used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 The analyst calculates surrogate standard recovery on all samples, blanks, and spikes. The analyst then determines if the recovery is within limits (limits established by performing QC procedures outlined in SOP 8000, Section 8.10).

8.3.1 If recovery is not with limits, the following is performed.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

- Reextract and reanalyze the sample if non of the above are a problem or flag the data as "estimated concentration."

8.4 <u>GC/MS confirmation</u>: Any compounds confirmed by two columns may also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory generated detection limits.

8.4.1 The GC/MS would normally require a minimum concentration of 10 ng/μL in the final extract, for each single-component compound.

8.4.2 The pesticide extract and associated blank are analyzed by GC/MS as per Section 7.0 of SOP 8270.

8.4.3 The confirmation may be from the GC/MS analysis of the base/neutral-acid extractables extracts (sample and blank). However, if the compounds are not detected in the vase/neutral-acid extract even through the concentration is high enough, a GC/MS analysis of the pesticide extract is performed.

8.4.4 A reference standard of the compound is also analyzed by GC/MS. The concentration of the reference standard is at a level that will demonstrate the ability to confirm the pesticides/PCBs identified by GC/ECD.

9.0 METHOD PERFORMANCE

9.1 The method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations. Concentrations used in the study ranged from 0.5 to 30 μg/L for single-component pesticides and from 8.5 to 400 μg/L for multi-component parameters. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix sample-preparation technique, optional cleanup techniques, and calibration procedures used.

Bibliography

1. U.S. EPA, "Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters, Category 10: Pesticides and PCBs," Report for EPA Contract 68-03-2605.
2. U.S. EPA, "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Environmental Monitoring and Support Laboratory, Cincinnati, OG 45268, October 1980.
3. Pressley, T.A., and J.E. Longbottom, "The Determination of Organohalide Pesticides and PCBs in Industrial and Municipal Wastewater: Method 617," U.S. EPA/EMSL, Cincinnati, Oh, EPA-600/4-84-006, 1982.

4. "Determination of Pesticides and PCB's in Industrial and Municipal Wastewaters, U.S. Environmental Protection Agency," Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, EPA-600/4-82-023, June 1982.
5. Goerlitz, D.F. and L.M. Law, Bulletin for Environmental Contamination and Toxicology, 6, 9, 1971.
6. Burke, J.A., "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.
7. Webb, R.G. and A.C. McCall, "Quantitative PCB Standards for Electron Capture Gas Chromatography," Journal of Chromatographic Science, 11, 366, 1973.
8. Miller, J.D., R.E. Thomas and H.J. Schattenberg, "EPA Method Study 18, Method 608: Organochlorine Pesticides and PCBs,"U.S. EPA.EmSL, Research Triangle Park, NC, EPA-600/4-84-061, 1984.
9. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
10. Provost, L.P., and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.
11. U.S. Food and Drug Association, Pesticides Analytical Manual (Volume 1), July 1985.
12. Sawyer, L.D, JAOAC, 56, 1015-1023 (1973), 61 272-281 (1978), 61 282-291 (1978).
13. Official Methods of Analysis of the Association of Official Analytical Chemists, 12th Edition; Changes in Methods, JAOAC 61, 465-466 (1978), Sec. 29.018.
14. Chemron Inc. Standard Operating Procedure 3580.
15. Chemron Inc. Standard Operating Procedure 3500.
16. Chemron Inc. Standard Operating Procedure 3600.
17. Chemron Inc. Standard Operating Procedure 8000.
18. Chemron Inc. Standard Operating Procedure 3510.
19. Chemron Inc. Standard Operating Procedure 3520.
20. Chemron Inc. Standard Operating Procedure 3540.
21. Chemron Inc. Standard Operating Procedure 3550.
22. Chemron Inc. Standard Operating Procedure 3620.
23. Chemron Inc. Standard Operating Procedure 3660.
24. Chemron Inc. Standard Operating Procedure 8270.
25. Chemron Inc. Standard Operating Procedure 8080.

Rev. 2.0

Date - December 7, 1994

Approved: _____ Date: 12/07/94

TABLE 1. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs[a]

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method Detection limit (ug/L)
Aldrin	2.40	4.10	0.004
α-BHC	1.35	1.82	0.003
β-BHC	1.90	1.97	0.006
δ-BHC	2.15	2.20	0.009
γ-BHC (Lindane)	1.70	2.13	0.004
Chlordane (technical)	e	e	0.014
4,4'-DDD	7.83	9.08	0.011
4,4'-DDE	5.13	7.15	0.004
4,4'-DDT	9.40	11.75	0.012
Dieldrin	5.45	7.23	0.002
Endosulfan I	4.50	6.20	0.014
Endosulfan II	8.00	8.28	0.004
Endosulfan sulfate	14.22	10.70	0.066
Endrin	6.55	8.10	0.006
Endrin aldehyde	11.82	9.30	0.023
Heptachlor	2.00	3.35	0.003
Heptachlor epoxide	3.50	5.00	0.083
Methoxychlor	18.20	26.60	0.176
Toxaphene	e	e	0.24
PCB-1016	e	e	nd
PCB-1221	e	e	nd
PCB-1232	e	e	nd
PCB-1242	e	e	0.065
PCB-1248	e	e	nd
PCB-1254	e	e	nd
PCB-1260	e	e	nd

[a]U.S. EPA. Method 617. Organochloride Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

e = Multiple peak response.

nd = not determined.

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 3. QC ACCEPTANCE CRITERIA[a]

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_S (%)
Aldrin	2.0	0.42	1.08-2.24	42-122
α-BHC	2.0	0.48	.98-2.44	37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4'-DDD	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.60	30-145
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50	12.7	27.8-55.6	41-126
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	38-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_S = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Parameter	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S' (ug/L)
Aldrin	0.81C+0.04	0.16$\bar{X}$-0.04	0.20$\bar{X}$-0.01
α-BHC	0.84C+0.03	0.13$\bar{X}$+0.04	0.23$\bar{X}$-0.00
β-BHC	0.81C+0.07	0.22$\bar{X}$+0.02	0.33$\bar{X}$-0.95
δ-BHC	0.81C+0.07	0.18$\bar{X}$+0.09	0.25$\bar{X}$+0.03
γ-BHC	0.82C-0.05	0.12$\bar{X}$+0.06	0.22$\bar{X}$+0.04
Chlordane	0.82C-0.04	0.13$\bar{X}$+0.13	0.18$\bar{X}$+0.18
4,4'-DDD	0.84C+0.30	0.20$\bar{X}$-0.18	0.27$\bar{X}$-0.14
4,4'-DDE	0.85C+0.14	0.13$\bar{X}$+0.06	0.28$\bar{X}$-0.09
4,4'-DDT	0.93C-0.13	0.17$\bar{X}$+0.39	0.31$\bar{X}$-0.21
Dieldrin	0.90C+0.02	0.12$\bar{X}$+0.19	0.16$\bar{X}$+0.16
Endosulfan I	0.97C+0.04	0.10$\bar{X}$+0.07	0.18$\bar{X}$+0.08
Endosulfan II	0.93C+0.34	0.41$\bar{X}$-0.65	0.47$\bar{X}$-0.20
Endosulfan Sulfate	0.89C-0.37	0.13$\bar{X}$+0.33	0.24$\bar{X}$+0.35
Endrin	0.89C-0.04	0.20$\bar{X}$+0.25	0.24$\bar{X}$+0.25
Heptachlor	0.69C+0.04	0.06$\bar{X}$+0.13	0.16$\bar{X}$+0.08
Heptachlor epoxide	0.89C+0.10	0.18$\bar{X}$-0.11	0.25$\bar{X}$-0.08
Toxaphene	0.80C+1.74	0.09$\bar{X}$+3.20	0.20$\bar{X}$+0.22
PCB-1016	0.81C+0.50	0.13$\bar{X}$+0.15	0.15$\bar{X}$+0.45
PCB-1221	0.96C+0.65	0.29$\bar{X}$-0.76	0.35$\bar{X}$-0.62
PCB-1232	0.91C+10.79	0.21$\bar{X}$-1.93	0.31$\bar{X}$+3.50
PCB-1242	0.93C+0.70	0.11$\bar{X}$+1.40	0.21$\bar{X}$+1.52
PCB-1248	0.97C+1.06	0.17$\bar{X}$+0.41	0.25$\bar{X}$-0.37
PCB-1254	0.76C+2.07	0.15$\bar{X}$+1.66	0.17$\bar{X}$+3.62
PCB-1260	0.66C+3.76	0.22$\bar{X}$-2.37	0.39$\bar{X}$-4.86

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{X}$, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in ug/L.

C = True value for the concentration, in ug/L.

$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

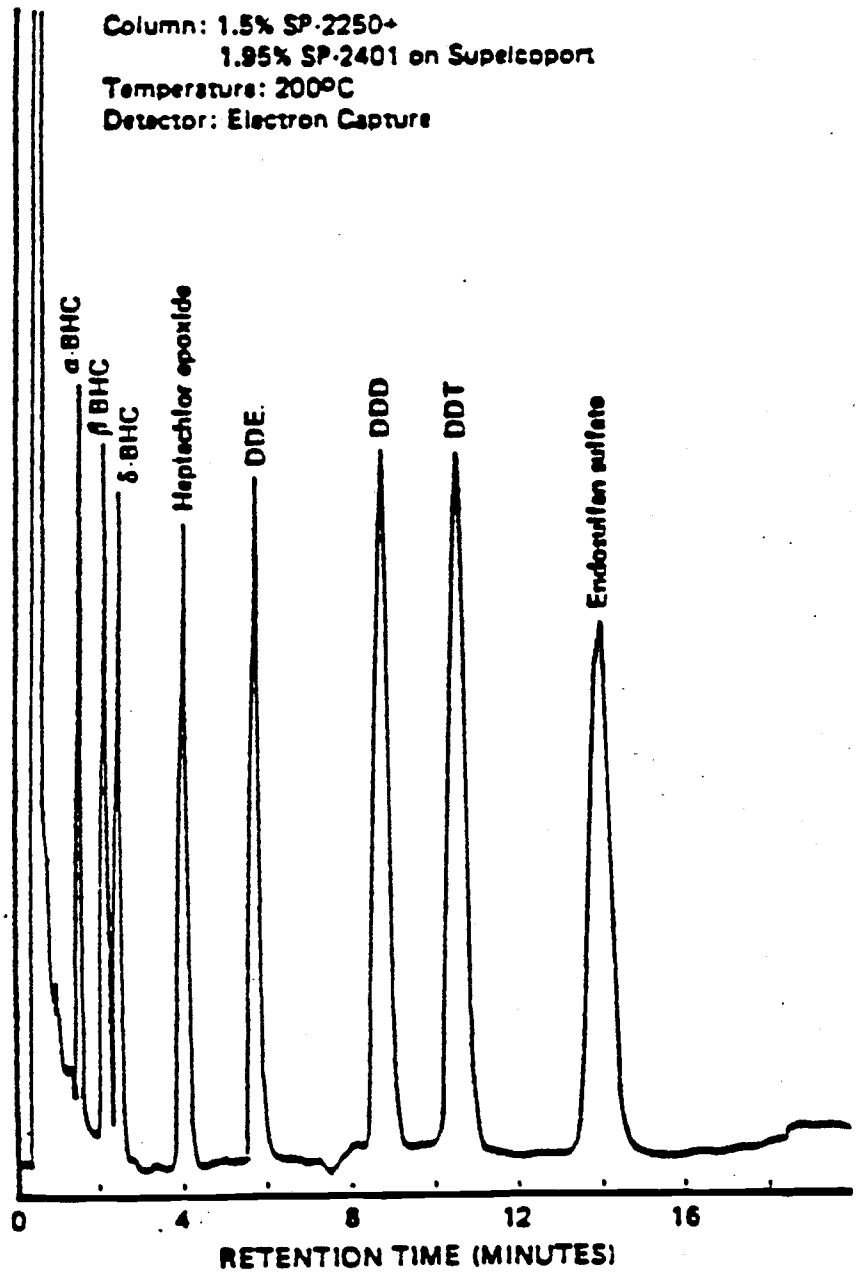


Figure 1. Gas chromatogram of pesticides.

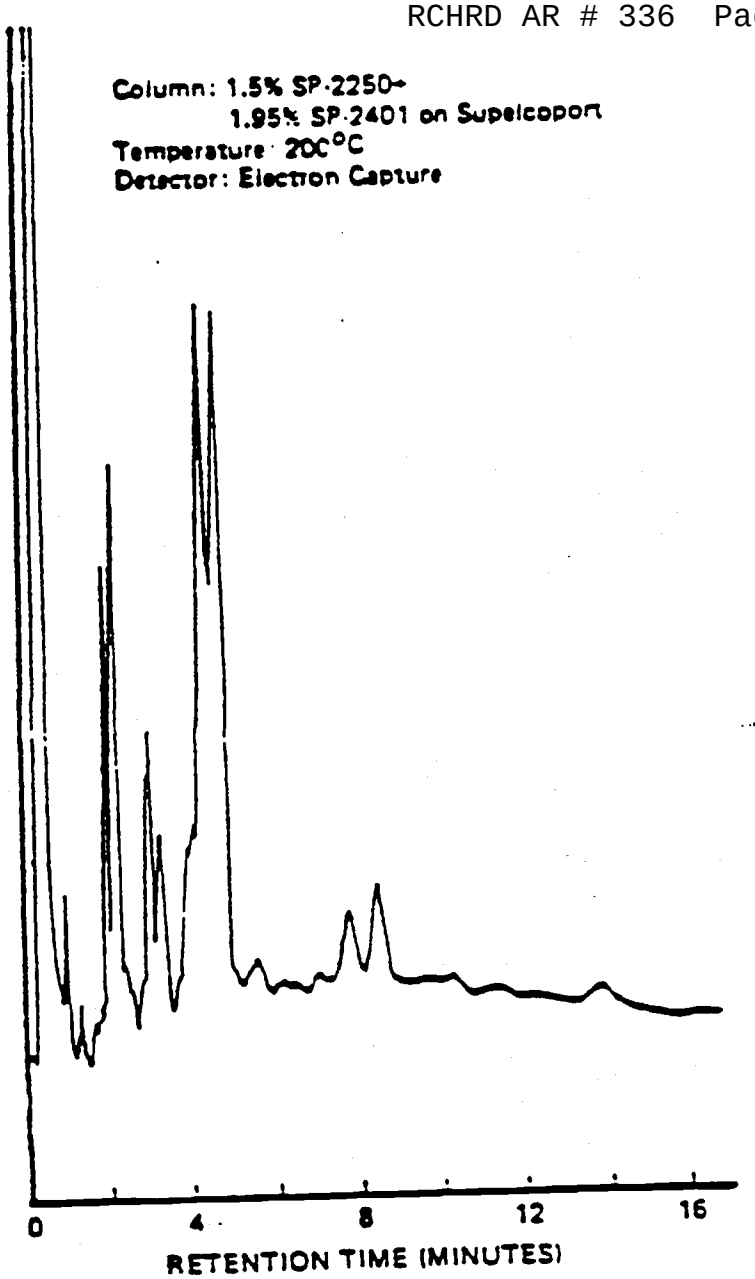


Figure 2. Gas chromatogram of chlordane.

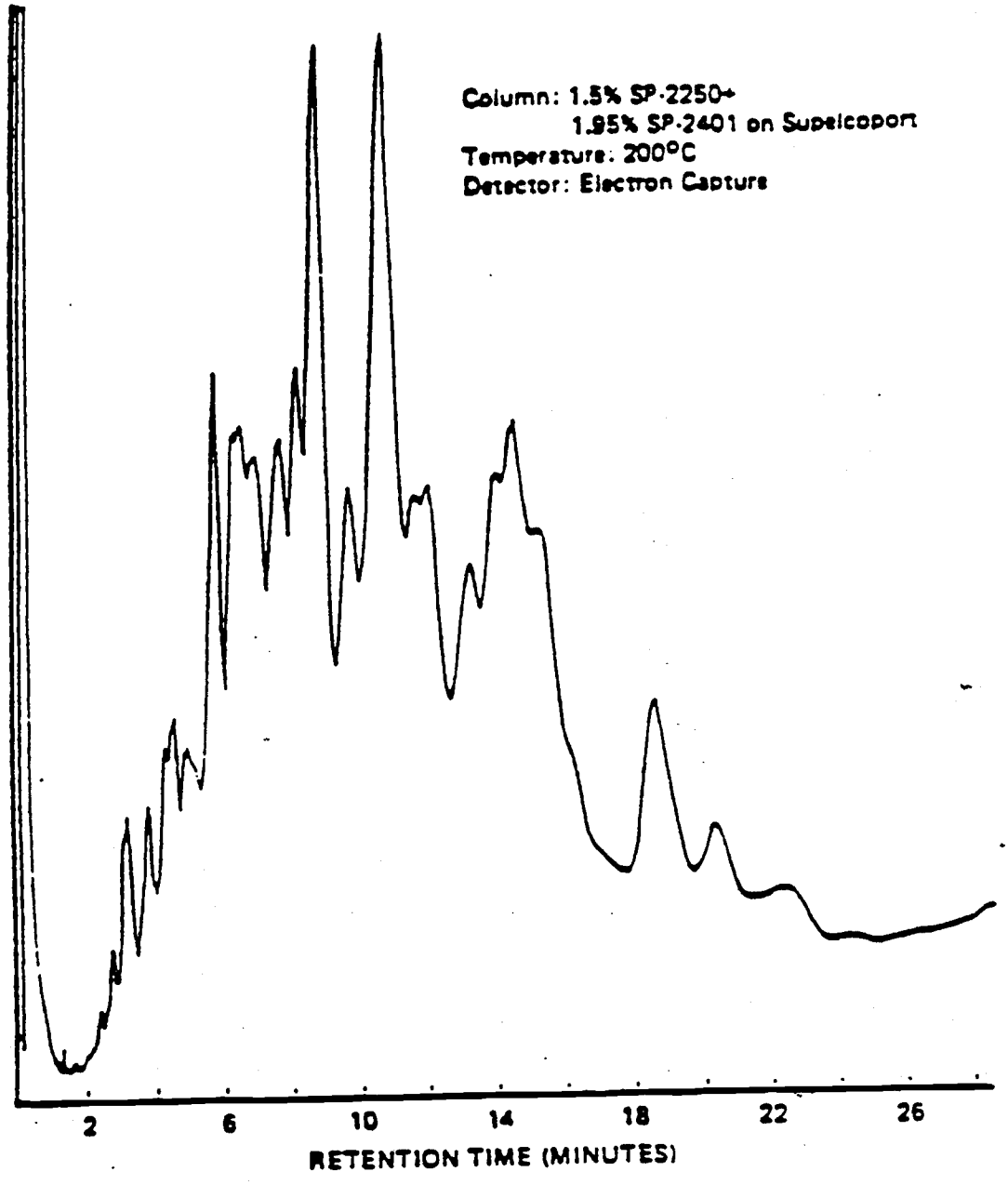


Figure 3. Gas chromatogram of toxaphene.

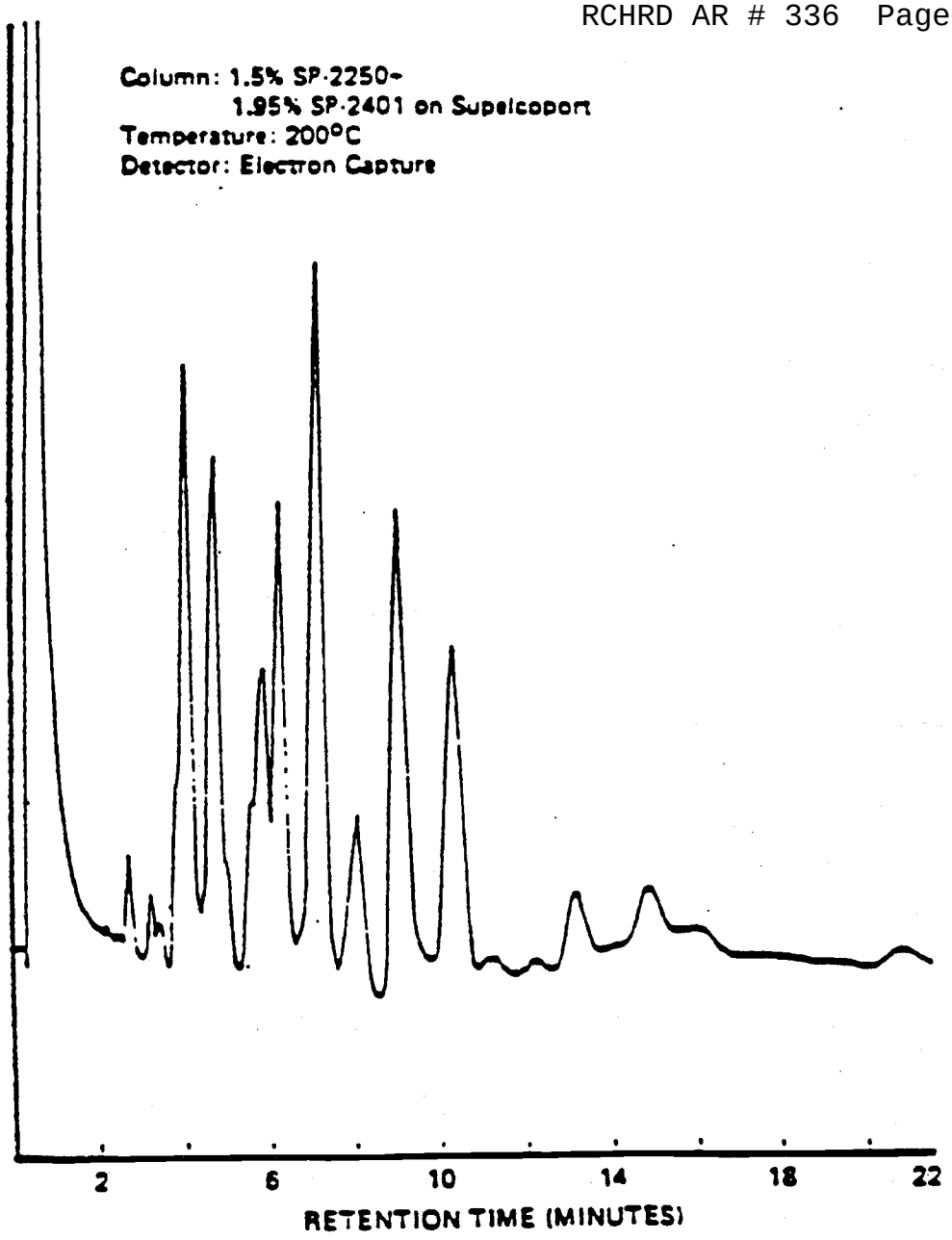


Figure 4. Gas chromatogram of PCB-1254.

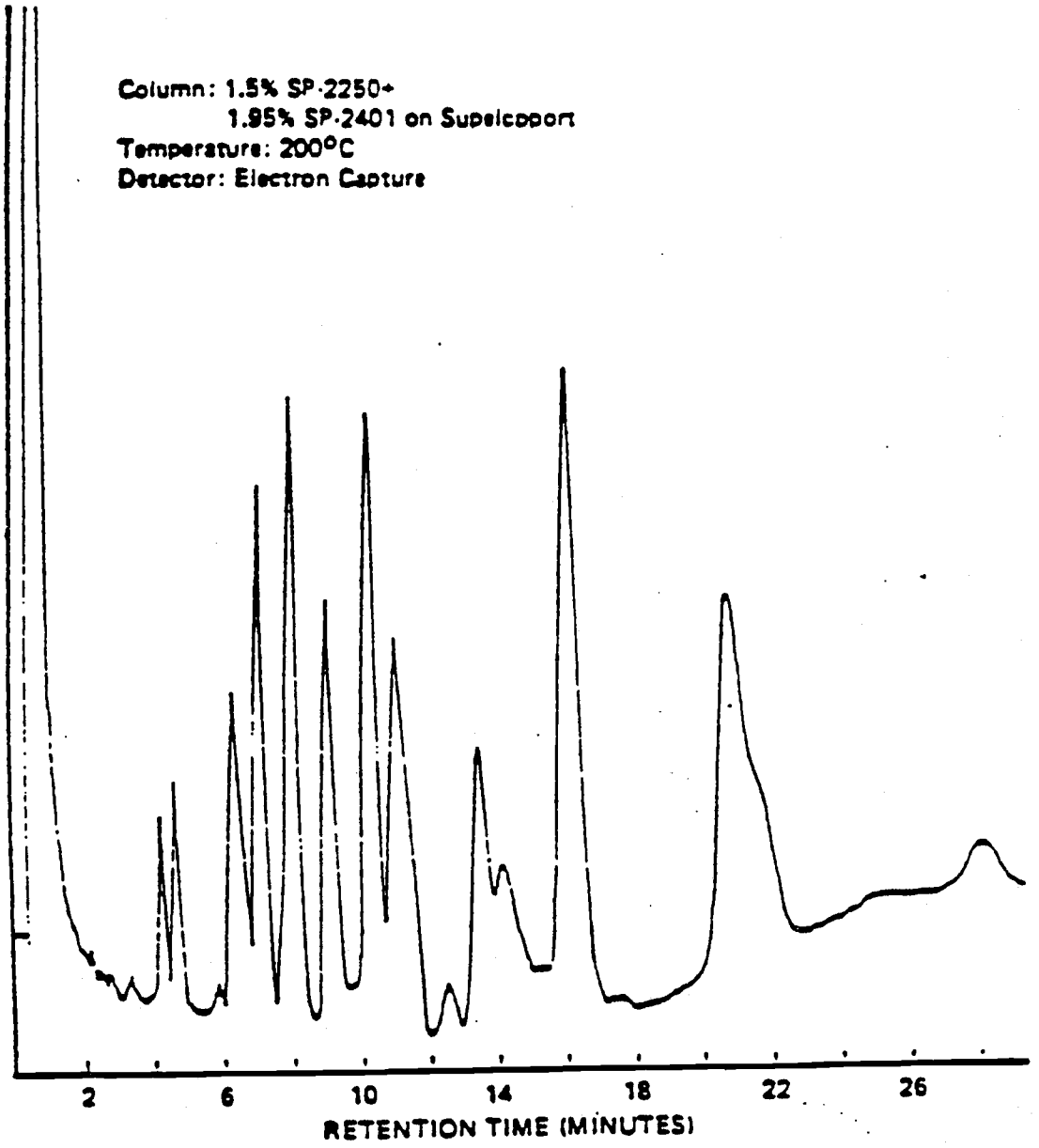


Figure 5. Gas chromatogram of PCB-1260.

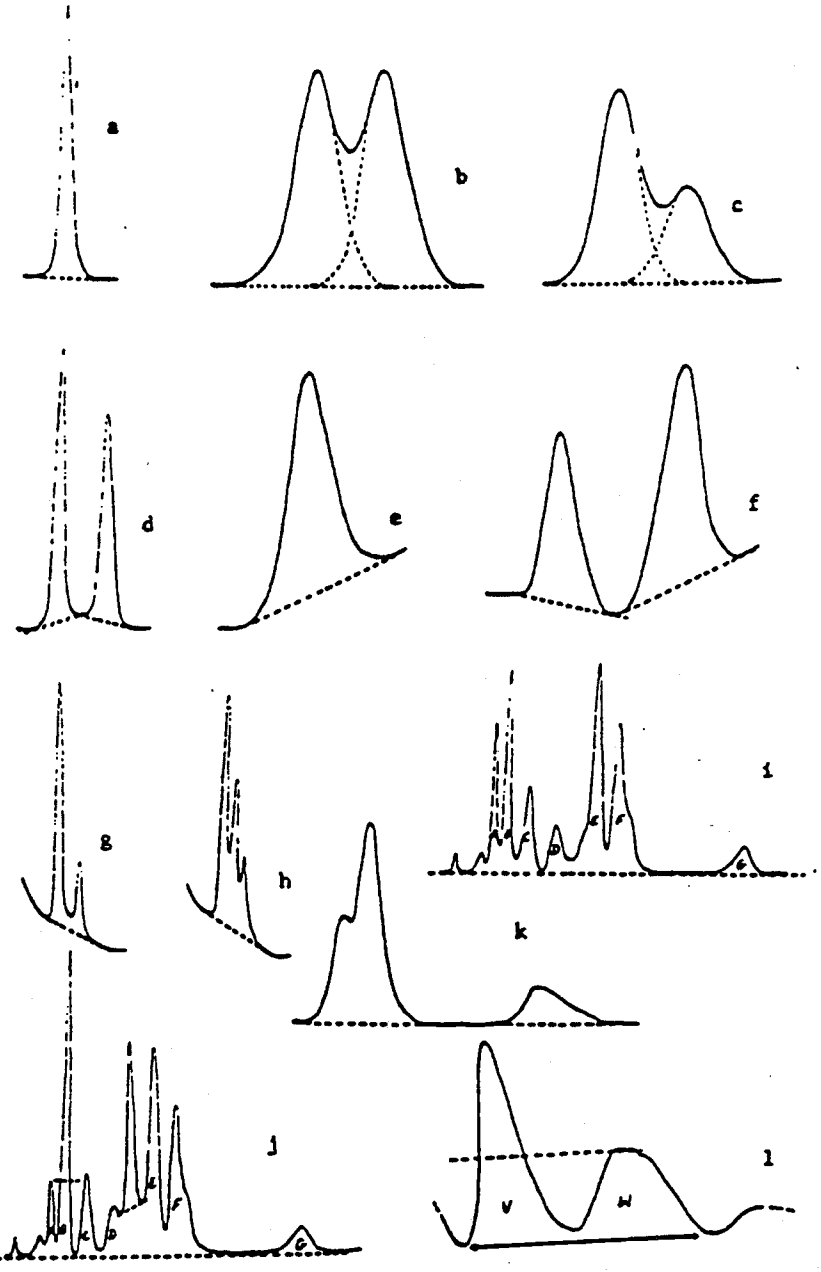


Fig. 6--Baseline construction for some typical gas chromatographic peaks. a, symmetrical separated flat baseline; b and c, overlapping flat baseline; d, separated (pen does not return to baseline between peaks); e, separated sloping baseline; f, separated (pen goes below baseline between peaks); g, α- and γ-BHC sloping baseline; h, α-, β-, and γ-BHC sloping baseline; i, chlordane flat baseline; j, heptachlor and heptachlor epoxide superimposed on chlordane; k, chair-shaped peaks, unsymmetrical peak; l, p,p'-DDT superimposed on toxaphene.

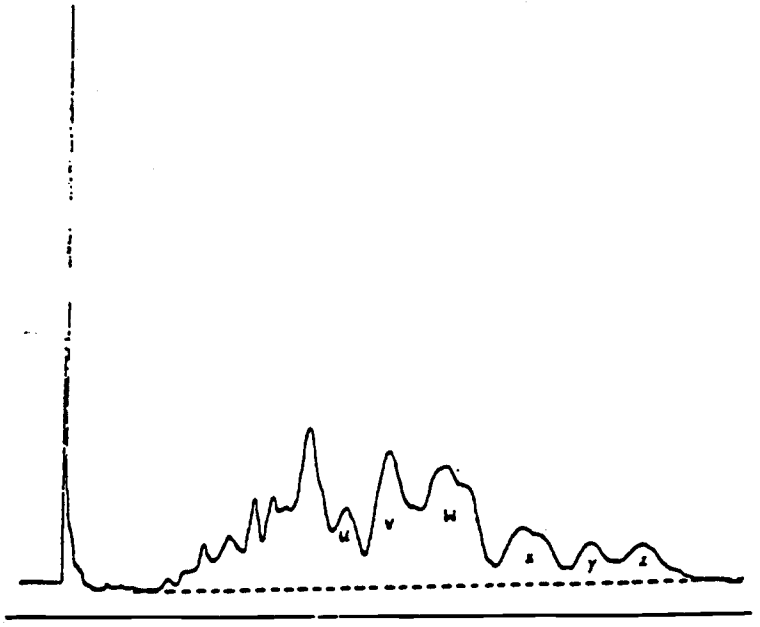


Fig. 7a--Baseline construction for multiple residues with standard toxaphene.

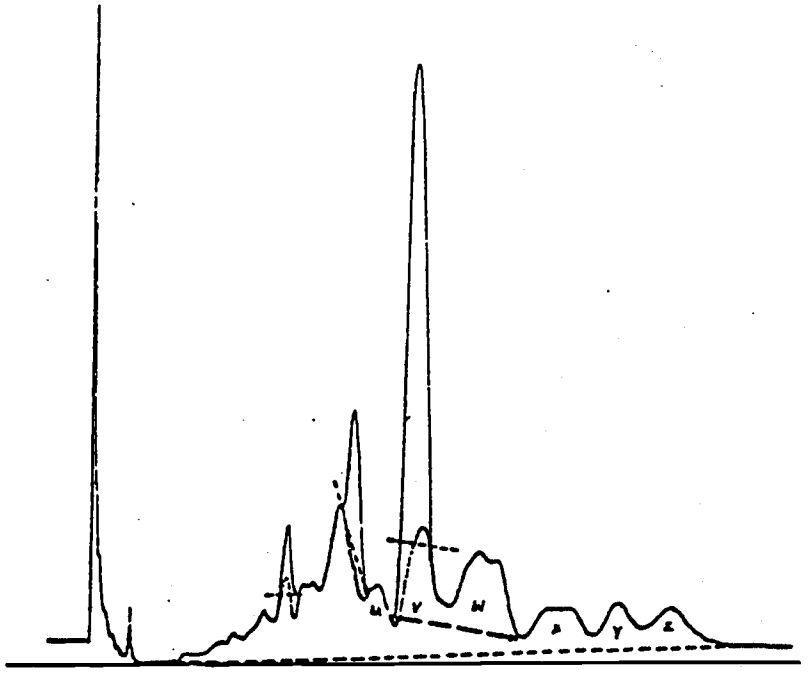


Fig. 7b--Baseline construction for multiple residues with toxaphene, DDE and o,p'-, and p,p'-DDT.

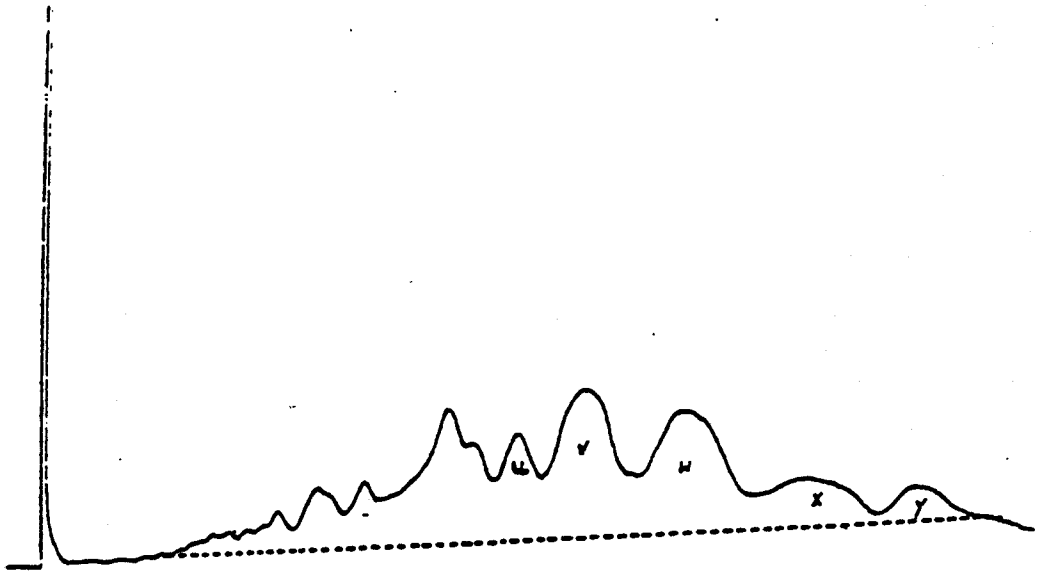


Fig. 8a--Baseline construction for multiple residues: standard toxaphene.

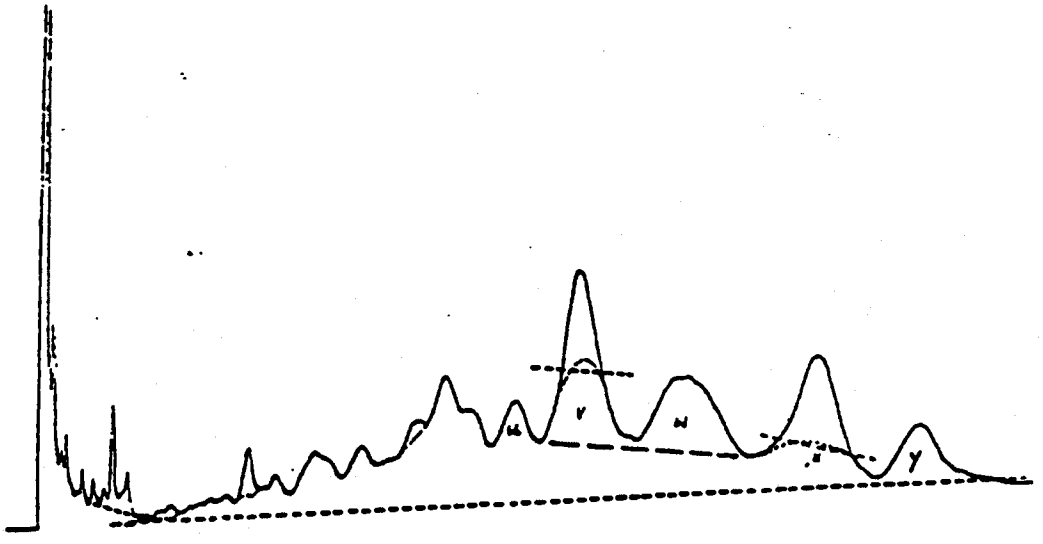


Fig. 8b--Baseline construction for multiple residues: rice bran with BHC, toxaphene, DDT, and methoxychlor.

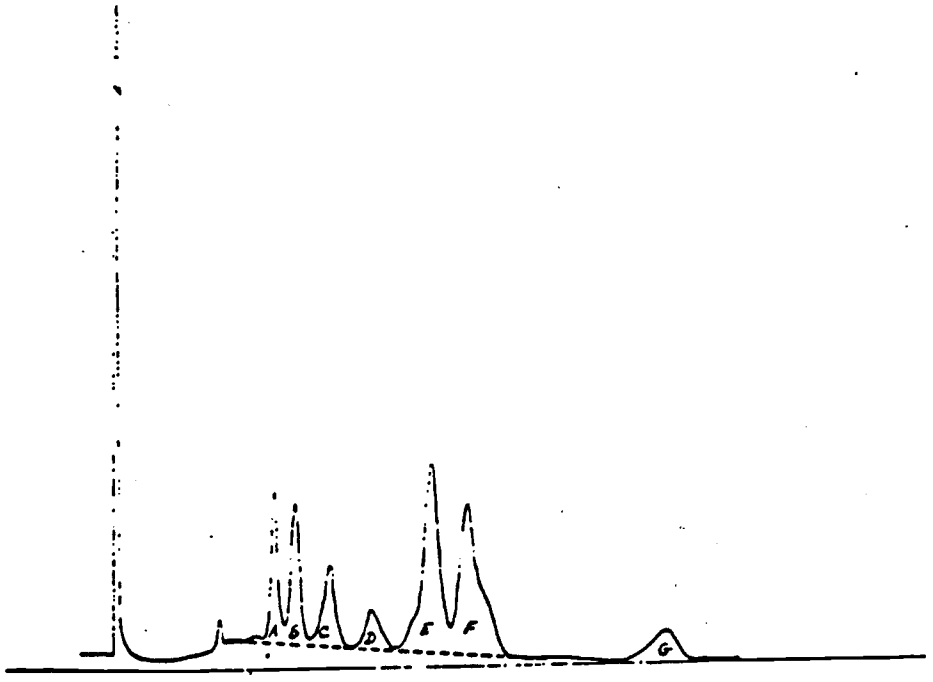


Fig. 9a--Baseline construction for multiple residues: standard chlordane.

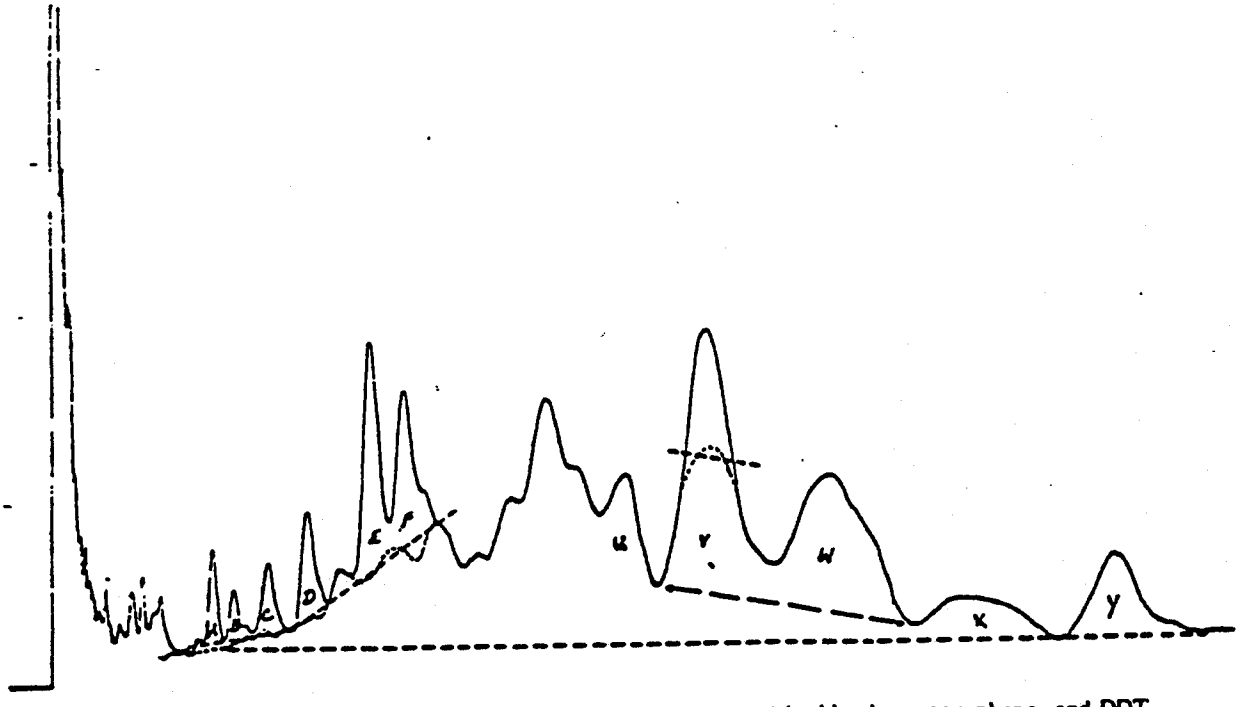
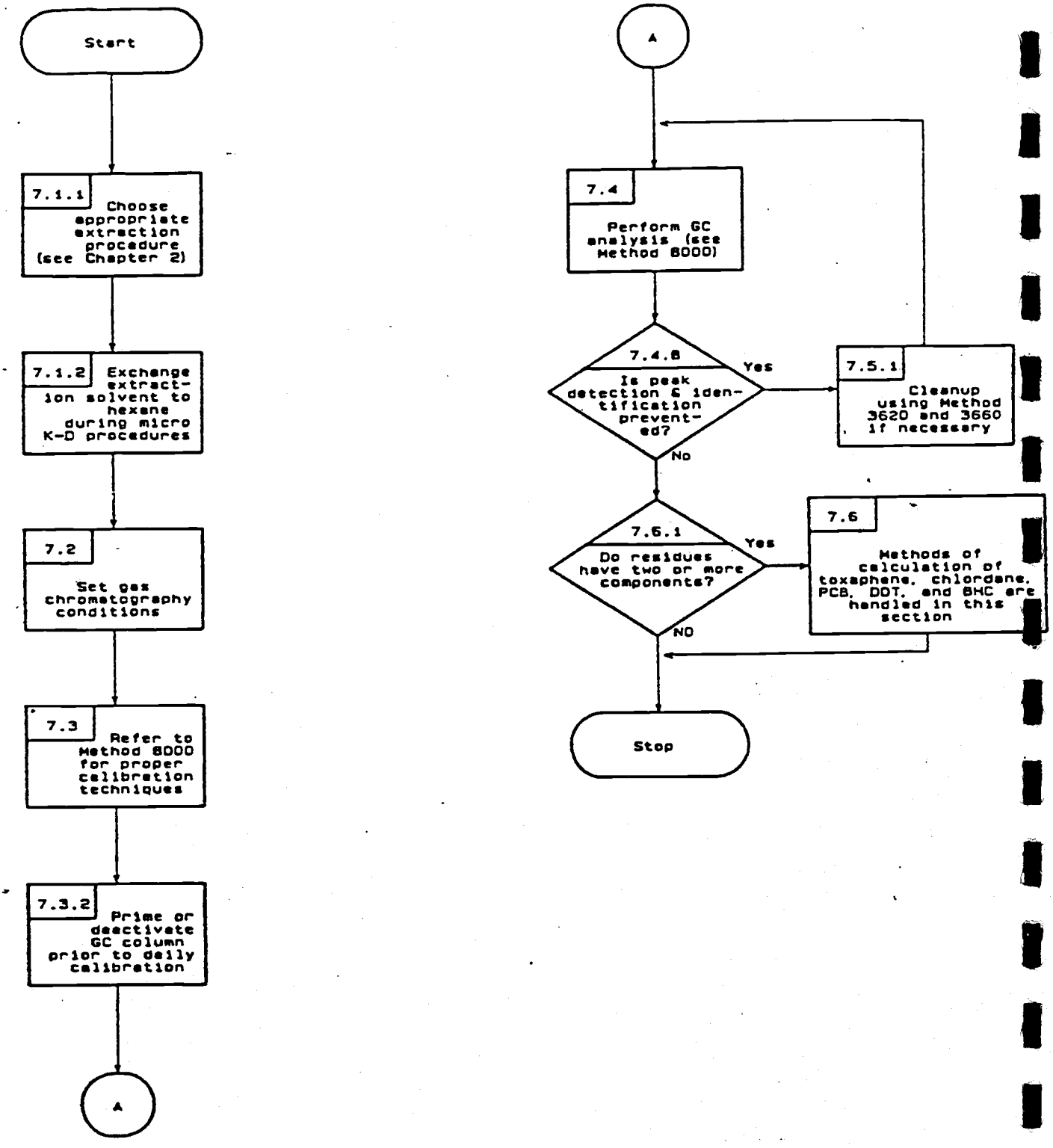


Fig. 9b--Baseline construction for multiple residues: rice bran with chlordane, toxaphene, and DDT.


Start
7.1.1 Choose appropriate extraction procedure (see Chapter 2)
7.1.2 Exchange extraction solvent to hexane during micro K-D procedures
7.2 Set gas chromatography conditions
7.3 Refer to Method 8000 for proper calibration techniques
7.3.2 Prime or deactivate GC column prior to daily calibration
A
A
7.4 Perform GC analysis (see Method 8000)
7.4.8 Is peak detection & identification prevented?
Yes
7.5.1 Cleanup using Method 3620 and 3660 if necessary
No
7.6.1 Do residues have two or more components?
Yes
7.6 Methods of calculation of toxaphene, chlordane, PCB, DDT, and BHC are handled in this section
NO
Stop

CHEMRON INC. - STANDARD OPERATING PROCEDURE 8150

CHLORINATED HERBICIDES

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 8150 is a gas chromatographic (GC) method for determining certain chlorinated acid herbicides. Table 1 indicates compounds that may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

1.2 When SOP 8150 is used to analyze unfamiliar samples, compound identification should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Section 8.4 provides gas chromatograph/mass spectrometer (GC/MS) criteria appropriate or the qualitative confirmation of compound identifications.

1.3 Only experienced analysts should be allowed to work with diazomethane due to the potential hazards associated with its use (the compound is explosive and carcinogenic).

2.0 SUMMARY OF METHOD

2.1 SOP 8150 provides extraction, esterification, and gas chromatographic conditions for the analysis of chlorinated acid herbicides. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivatizing agent. After excess reagent is removed, the esters are determined by gas chromatography employing an electron capture detector, microcoulometric detector, or electrolytic conductivity detector (Goerlitz and Lamar, 1967). The results are reported as the acid equivalents.

2.2 The sensitivity of SOP 8150 usually depends on the level of interferences rather than on instrumental limitations.

3.0 INTERFERENCES

3.1 Refer to SOP 8000.

3.2 Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols, including chlorophenols, may also interfere with this procedure.

3.3 Alkaline hydrolysis and subsequent extraction of the basic solution remove many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis.

3.4 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware and glass wool must be acid-rinsed, and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

4.1.1 Columns:

4.1.1.1 Column 1a and 1b: 1.8-m x 4-mm I.D. glass, packed with 1.5% SP-2250 on Supelcort (100/120).

4.1.1.2 Column 2: 1.8-m x 4-m I.D. glass, packed with 5% OV-210 on Gas Crom Q (100/120 mesh).

4.1.1.3 Column 3: 1.98-m x 2-mm I.D. glass, packed with 0.1% SP-1000 on 80/100 mesh Carbopack C.

4.1.2 Detectors: Electron capture (ECD)

4.2 Erlenmeyer flasks: 250- and 500-mL Pyrex, with 24/40 ground-glass joint.

4.3 Beaker: 500-mL.

4.4 Diazomethane generator: Refer to Section 7.3 to determine which method of diazomethane generation should be used for a particular application.

4.4.1 Diazald kit: recommended for the generation of diazomethane using the procedure given in Section 7.3.2 (Aldrich Chemical Co., Cat. No 210,025-2).

4.4.2 Assemble from two 20 x 150-mm test tubes, two Neoprene rubber stoppers, and a source of nitrogen. Use Neoprene rubber stoppers with holes drilled in them to accommodate glass delivery tubes. The exit tube must be drawn to a point to bubble diazomethane through the sample extract. The generator assembly is shown in Figure 1. The procedure for use of this type of generator is given in Section 7.3.3.

4.5 Vials: Amber glass, 10- to 15-mL capacity with Teflon-lined screw cap.

4.6 Separatory funnel: 2-L 125-mL, and 60-mL.

4.7 Drying column: 400-mm x 20-mm I.D. Pyres chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.8 Kuderna-Danish (K-D) apparatus:

4.2.1 Concentrator tube: 10-mL, graduated (Kontes K-570050-1025). Ground-glass stopper is used to prevent evaporation of extracts.

4.2.2 Evaporation flask: 500-mL (Kontes K-570001-500). Attach to concentrator tube with springs.

4.2.3 Snyder column: Three-ball macro (Kontes K-503000-0121).

4.2.4 Snyder column: Two-ball micro (Kontes K-569001-0219).

4.9 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide).

4.10 Water bath: Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.11 Microsyringe: 10-μL.

4.12 Wrist shaker: Burrell Model 75.

4.13 Glass wool: Pyrex, acid washed.

4.14 Balance: Analytical, capable of accurately weighting to the nearest 0.0001 g.

4.15 Syringe: 5-mL.

4.16 Glass rod.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.2 Sulfuric acid solution:

5.2.1 (1:1) (V/V) - Slowly add 50 mL of H_2SO_4 (sp. gr. 1.84) to 50 mL or reagent water.

5.2.2 (1:1) (V/V) - Slowly add 25 mL of H_2SO_4 (sp. gr. 1.84) to 50 mL or reagent water.

5.3 Hydrochloric acid: (ACS), (1:9) (V/V) - add one volume of concentrated HCl to 9 volumes of reagent water.

5.4 Potassium hydroxide solution: 37% aqueous solution (W/V). Dissolve 37 g ACS grade potassium hydroxide pellets in reagent water and dilute to 100 mL.

5.5 Carbitol (Diethylene glycol monoethyl ether): (ACS), available from Aldrich Chemical Co.

5.6 Solvents:

5.6.1 Acetone, methanol, ethanol, methylene chloride, hexane (pesticide quality).

5.6.2 Diethyl ether: Pesticide quality or equivalent. Must be free of peroxides, as indicated by EM Quant test strips (available from Scientific Products Co., Cat. No. P1126-8, and other suppliers). Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL ethanol preservative must be added to each liter of ether.

5.7 Sodium sulfate: (ACS) granular, acidified, anhydrous. Heat in a shallow tray at 400°C for a minimum of 4 hr to remove phthalates and other interfering organic substances. Alternatively, heat 16 hr at 400-500°C in a shallow tray or Soxhlet extract with methylene chloride for 48 hr. Acidify by slurrying 100 g sodium sulfate with enough diethyl ether to just cover the solid; then add 0.1 mL of concentrated sulfuric acid and mix thoroughly. Remove the ether under a vacuum. Mix 1 g of the resulting solid with 5 mL of reagent water and measure the pH of the mixture. It must be below a pH of 4. Store at 130°C.

5.8 N-Methyl-N-nitros-p-toluenesulfonamide (Diazald): (ACS) available from Aldrich Chemical Co.

5.9 Silicic acid: chromatographic grade, nominal 100 mesh. Store at 130°C.

5.10 Stock standard solutions:

5.10.1 Prepare stock stand solutions at a concentration of 1.00 ug/uL by dissolving 0.0100 g of assayed reference material in isoctane and diluting to volume in a 10-mL volumetric flask. A small volume of toluene may be necessary to put some pesticides in solution. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.10.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.10.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.11 Calibration standards: Calibration standards at a minimum of five concentration levels for each parameter of interest are prepared through dilution of the stock standards with isooctane. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in the real samples or should define

the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

5.12 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.12.1 Prepare calibration standards at a minimum of five concentration levels for each analyte of interest as described in Paragraph 5.3.

5.12.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isoctane.

5.12.3 Analyze each calibration standard according to Section 7.0.

5.13 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with one or two herbicide surrogates (e.g., herbicides that are not expected to be present in the sample) recommended to encompass the range of the temperature program used in this method. Deuterated analogs of analytes should not be used as surrogates for gas chromatographic analysis due to coelution problems.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Preparation of solid samples:

7.1.1 Extraction:

7.1.1.1 To a 500-mL, wide-mouth Erlenmeyer flask add 50 g (dry weight) of the well-mixed, moist solid sample. Adjust to pH to 2 with concentrated GCl and monitor the pH for 15 min with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

7.1.1.2 Add 20 mL acetone to the flask and mix the contents with the wrist shaker for 20 min. Add 80 mL diethyl ether to the same flask and shake again for 20 min. Decant the extract and measure the volume of solvent recovered.

7.1.1.3 Extract the sample twice more using 20 mL of acetone followed by 80 mL of diethyl ether. After addition of each solvent, the mixture should be shaken with the wrist shaker for 10 min and the acetone-ether extract decanted.

7.1.1.4 After the third extraction, the volume of extract recovered should be at least 75% of the volume of added solvent. If this is not the case, additional extractions may be necessary. Combine the extracts in a 2-liter separatory funnel containing 250 mL of 5% acidified sodium sulfate. If an emulsion forms, slowly add 5 g of acidified sodium sulfate (anhydrous) until the solvent-water mixture separates. A quantity of acidified sodium sulfate equal to the weight of the sample may be added, if necessary.

7.1.1.5 Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for 1 min and allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract phase (top layer) in a 500-mL ground-glass Erlenmeyer flask. Place the aqueous phase back into the separatory funnel and re-extract using 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in the 500-mL Erlenmeyer flask.

7.1.2 Hydrolysis:

7.1.2.1 Add 30 mL of reagent water, 5 mL of 37% KOH, and one or two clean boiling chips to the flask. Place a three-ball Snyder column on the flask, evaporate the diethyl ether on a water bath, and continue to heat for a total of 90 min.

7.1.2.2 Remove the flask from the water bath and allow to cool. Transfer the water solution to a 125-mL separatory funnel and extract the basic solutions once with 40 mL and then twice with 20 mL of diethyl ether. Allow sufficient time for the layers to separate and discard the ether layer each time. The phenoxy acid herbicides remain soluble in the aqueous phase as potassium salts.

7.1.3 Solvent cleanup:

7.1.3.1 Adjust the pH to 2 by adding 5 mL cold (4°C) sulfuric acid (1:3) to the separatory funnel. Be sure to check the pH at this point. Extract the herbicides once with 40 mL and twice with 20 mL of diethyl ether. Discard the aqueous phase.

7.1.3.2 Combine ether extracts in a 125-mL Erlenmeyer flask containing 1.0 g of acidified anhydrous sodium sulfate. Stopper and allow the extract to remain in contact with the acidified sodium sulfate. If concentration and esterification are not to be performed immediately, store the sample over night in the refrigerator.

7.1.3.2 Combine ether extracts in a 125-mL Erlenmeyer flask containing 1.0 g of acidified anhydrous sodium sulfate. Stopper and allow the extract to remain in contact with the acidified sodium sulfate. If concentration and esterification are not to be performed immediately, store the sample overnight in the refrigerator.

7.1.3.3 Transfer the ether extract, through a funnel plugged with acid-washed glass wool, into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Use a glass rod to crush caked sodium sulfate during the transfer. Rinse the Erlenmeyer flask and column with 20-30 mL of diethyl ether to complete the quantitative transfer.

7.1.3.4 Add one or two clean boiling chips to the flask and attack a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of diethyl ether to the top. Place the apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 15-20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reached 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min.

7.1.3.5 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of diethyl ether. A 5-mL syringe is recommended for this operation. Add a fresh boiling chip, attach a micro-Snyder column to the concentrator tube, and prewet the column by adding 0.5 mL of ethyl ether to the top. Place the micro-K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot

water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5-10 min. When the apparent volume of the liquid reaches 0.5 mL, remove the micro-K-D from the bath and allow it to drain and cool. Remove the Snyder column and add 0.1 mL of methanol. Rinse the walls of the concentrator tube while adjusting the extract volume to 1.0 mL with deithyl ether. Proceed to Section 7.3 for esterification.

7.2 Prepare of liquid samples:

7.2.1 Extraction:

7.2.1.1 Mark the water miniscus on the side of the sample container for later determination of sample volume. Pour the entire sample into a 2-liter seperatory funnel and check the pH with wide-range pH paper. Adjust the pH to less than 2 with sulfuric acid (1:1).

7.2.1.2 Add 150 mL of diethyl ether to the sample bottle, seal, and shake for 30 sec to rinse the walls. Transfer the solvent wash to the seperatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 min. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Drain the aqueous phase into a 1-liter Erlenmeyer flask. Collect the solvent extract in a 250-mL ground-glass Erlenmeyer flask containing 2 mL of 37% KOH. Approximately 80 mL of the diethyl ether will remain dissolved in the aqueous phase.

7.2.1.3 Repeat the extraction two more times using 50 mL of diethyl ether each time. Combine the extracts in the Erlenmeyer flask. (Rinse the 1-liter flask with each additional aliquot of extracting solvent.)

7.2.2 Hydrolysis:

7.2.2.1 Add one or two clean boiling chips and 15 mL of reagent water to the 250-mL flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of diethyl ether to the top of the column. Place the apparatus on a hot water bath (60-65°) so that the bottom of the flask is bathed with hot water vapor. Although the diethyl ether will evaporate in about 15 min, continue heating for a total of 60 min, beginning

from the time the flask is placed in the water bath. Remove the apparatus and let stand at room temperature for at least 10 min.

7.2.2.2 Transfer the solution to a 60-mL separatory funnel using 5-10 mL of reagent water. Wash the basic solution twice by shaking for 1 min with 20-mL portions of diethyl ether. Discard the organic phase. The herbicides remain in the aqueous phase.

7.2.3 Solvent cleanup:

7.2.3.1 Acidify the contents of the separatory funnel to pH 2 by adding 2 mL of cold (4°C) sulfuric acid (1:3). Test with pH indicator paper. Add 20 mL diethyl ether and shake vigorously for 2 min. Drain the aqueous layer into a 250-mL Erlenmeyer flask, and pour the organic layer into a 125-mL Erlenmeyer flask containing about 0.5 g of acidified sodium sulfate. Repeat the extraction twice more with 10-mL flask. Allow the extract to remain in contact with the sodium sulfate for approximately 2 hr.

7.2.3.2 Transfer the ether extract, through a funnel plugged with acid-washed glass wool, into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Use a glass rod to crush caked sodium sulfate during the transfer. Rinse the Erlenmeyer flask and column with 20-30 mL of diethyl ether to complete the quantitative transfer.

7.2.3.3 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of diethyl ether to the top. Place the apparatus on a hot water bath (60-65°) so that the concentrator tube is partially immersed in the hot water and the entire low rounded surface of the flask is bathed in vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 15-20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reached 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min.

7.2.3.4 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of diethyl ether. A 5-mL syringe is recommended for this operation. Add a fresh boiling chip, attach a micro-Snyder column to the concentrator tube, and prewet the column by adding 0.5 mL of ethyl ether to the top. Place the micro-K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot

water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5-10 min. When the apparent volume of the liquid reaches 0.5 mL, remove the micro-K-D from the bath and allow it to drain and cool. Remove the Snyder column and add 0.1 mL of methanol. Rinse the walls of the concentrator tube while adjusting the extract volume to 1.0 mL with deithyl ether.

7.2.3.5 Determine the original sample volume by refilling the sample bottle to the mark with water and transferring to a 1-liter graduated cylinder. Record the sample volume to the nearest 5 mL.

7.3 Esterification:

7.3.1 Two methods may be sued for the generation of diazomethane: the bubbler method (set up shown in Figure 1) and the Diazald kit method. The bubbler method is suggested when small batches (10-15) of samples require esterification. The bubbler method works well with samples that have low concentrations of herbicides (e.g., aqueous samples) and is safer to use than the Diazald kit procedure. The diazald kit method is good for large quantities of samples needing esterification. The diazald kit method is more effective than the bubbler method for soils or samples that may contain high concentrations of herbicides (e.g., samples such as soils that may result in yellow extracts following hydrolysis may e difficult to handle by the bubbler method). The diazomethane derivatization (U.S. EPA, 1971) procedures, described below, will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use. The following precautions should be taken:

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

- Use a safety screen.
- Use mechanical pipetting aides.
- Do not heat above 90°C -- EXPLOSION may result.
- A void grinding surfaces, ground-glass joints, sleeve bearings, glass stirrers -- EXPLOSION may result.
- Store away rom alkali metals -- EXPLOSION may result.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

7.3.2 Diazald kit method: Instructions for preparing diazomethane are provided with the generator kit.

7.3.2.1 Add 2 mL of diazomethane solution and let sample stand for 10 min with occasional swirling.

7.3.2.2 Rinse inside wall of ampule with several hundred μL of diethyl ether. Allow solvent to evaporate spontaneously at room temperature to about 2 mL.

7.3.2.3 Dissolve the residue in 5 mL of hexane. Analyze by gas chromatography.

7.3.3 Bubbler method: Assemble the diazomethane bubbler (see Figure 1)

7.3.3.1 Add 5 mL of diethyl ether to the first test tube. Add 1 mL of diethyl ether, 1 mL of carbitol, 1.5 mL of 37% KOH, and 0.1-0.2 g Diazald to the second test tube. Immediately place the exit tube into the concentrator tube containing the sample extract. Apply nitrogen flow (10 mL/min) to bubble diazomethane through the extract for 10 min or until the yellow color of diazomethane persists. The amount of Diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1-0.2 g of Diazald may e added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 min of total esterification.

7.3.3.2 Remove the concentrator tube and seal it with a Neoprene or Teflon stopper. Store at room temperature in a hood for 20 min.

7.3.3.3 Destroy any unreacted diazomethane by adding 0.1-0.2 g silicic acid to the concentrator tube. Allow to stand until the evolution of nitrogen gas has stopped. Adjust the sample volume to 10.0 mL with hexane. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. It is recommended that the methylated extracts be analyzed immediately to minimize the trans-esterification and other potential reactions that may occur. Analyze by gas chromatography.

7.4 Gas chromatography conditions (Recommended):

7.4.1 Column 1a: Set 5% methane/95% argon carrier gas flow at 70 mL/min flow rate. Column temperature is set at 185°C isothermal.

7.4.2 Column 1b: Set 5% methane/95% argon carrier gas flow at 70 mL/min flow rate. Column temperature is set at 140°C for six min and then programmed at 10°C/min to 200°C and held.

7.4.3 Column 2: Set 5% methane/95% argon carrier gas flow at 70 mL/min flow rate. Column temperature is set at 185°C isothermal.

7.4.4 Column 3: Set nitrogen (ultra-high purity) carrier gas at 25-mL/min flow rate. Column temperature is set at 200°C and then immediately programmed at 10°C/min to 150°C and held.

7.5 Calibration: Refer to SOP 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.5.1 The procedure for internal or external calibration may be used. Refer to SOP 8000 for a description of each of these procedures.

7.5.2 The following gas chromatographic columns are recommended for the compounds indicated:

Parameter	Column
Dicamba	1a,2
2,4-D	1a,2
2,4,5-TP	1a,2
2,4,5-T	1a,2
2,4-DB	1a
Dalapon	3
MCPP	1b
MCPA	1b
Dichloroprop	1b
Dinoseb	1b

7.6 Gas chromatographic analysis:

7.6.1 Refer to SOP 8000. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to injection.

7.6.2 Follow Section 7.6 in SOP 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows,and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.6.3 Examples of chromatograms for various chlorophenoxy herbicides are shown in Figures 2 through 4.

7.6.4 Record the sample volume injected and the resulting peak sized (in area units or peak heights).

7.6.5 Using either the internal or external calibration procedure (SOP 8000), determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration proposes.

7.6.6 If calibration standards have been analyzed in the same manner as the samples (e.g., have undergone hydrolysis and esterification), then the calculation of concentration given in SOP 8000, Section 7.8 should be used. However, if calibration is done using standards made from methyl ester compounds (compounds not esterified by application of this method), then the calculation of concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide.

7.6.7 If peak detection and identification are prevented due to interferences, further cleanup is required. Before using any cleanup procedure, the analyst must process a series of standards through the procedure to validate elution patterns and the absence of interferences from reagents.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in SOP 3600 and in the specific cleanup method.

8.2 Procedures to check the GC system operation are found in SOP 8000, Section 8.6.

8.2.1 Select a representative spike concentrations for each compound (acid or ester) to be measured. Using stock standards, prepare a quality control check sample concentrate in acetone 1,000 times more concentrated than the selected concentrations.

8.2.2 Table 3 indicates Operator Accuracy and Precision for this method. Compare the results obtained with the results given in Table 3 to determine if the data quality is acceptable.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in SOP 8000, Section 8.10).

8.3.1 If recovery is not with limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

- Reextract and reanalyze the sample if non of the above are a problem or flag the data as "estimated concentration."

8.4 GC/MS confirmation: Any compounds confirmed by two columns may also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory generated detection limits.

8.4.1 The GC/MS would normally require a minimum concentration of 10 ng/μL in the final extract, for each single-component compound.

8.4.2 When available, chemical ionizaiton mass spectra may be employed to aid the qualitative identification process.

8.4.3 Should these MS procedures fail to provide satisfactory results, additional steps may be taken before reanalysis. These steps may include the use of alternate packed or capillary GC columns or additional cleanup.

9.0 METHOD PERFORMANCE

9.1 In a single laboratory, using reagent water and effluent from publicly owned treatment works (POTW), the average recoveries presented in Table 3 were obtained. The standard deviations of the percent recoveries of these measurements are also included in Table 3.

Bibliography

1. U.S. EPA, National Pollutant Discharge Elimination System, Appendix A, Fed. Reg., 38, No. 75, Pt. II, Method for Chlorinated Phenoxy Acid Herbicides in industrial Effluent, Cincinnati, Ohio, 1971.
2. Goerlitz, D.G. and W.L. Lamar, Determination of Phenoxy Acid Herbicides in Water by Electron Capture and Microcoulometric Gas Chromatogram," U.S. Geol. Survey Water Supply Paper, 1817-C, 1967.
3. Burke, J.A., "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.
4. U.S. EPA, Extraction and Cleanup Procedure for the Determination of Phenoxy Acid Herbicides in Sediment," EPA Toxicant and Analysis Center, Bay St. Louis, Mississippi, 1972.
5. "Pesticide Methods Evaluation," Letter Report #33 for EPA Contract No. 68-03-2697. Available from U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.
6. McNair, H.M. and E.J. Bonelli, "Basic Chromatography," Consolidated Printing, Berkeley, California, p. 52, 1969.
7. Eichelberger, J.W., L.E. Harris, and W.L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry," Analytical Chemistry, 47, 995, 1975.
8. Glaser, J.A. et.al., "Trace Analysis for Wastewaters," Environmental Science & Technology, 15, 1426, 1981.
9. U.S. EPA, "Method 615. The Determination of Chlorinated Herbicides in Industrial and Municipal Wastewater," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 44268, June 1982.
10. Chemron Inc. Standard Operating Procedure 8000.
11. Chemron Inc. Standard Operating Procedure 3500.
12. Chemron Inc. Standard Operating Procedure 3600.

Rev. 2.0

Date - December 7, 1994

Approved: _____ Date: 12/07/94

TABLE 1.
CHROMATOGRAPHIC CONDITIONS AND DETECTION LIMITS
FOR CHLORINATED HERBICIDES

Compound	Retention time (min)[a]				Method detection limit (µg/L)
	Col.1a	Col.1b	Col.2	Col.3	
2,4-D	2.0	-	1.6	-	1.2
2,4-DB	4.1	-	-	-	0.91
2,4,5-T	3.4	-	2.4	-	0.20
2,4,5-TP (Silvex)	2.7	-	2.0	-	0.17
Dalapon	-	-	-	5.0	5.8
Dicamba	1.2	-	1.0	-	0.27
Dichloroprop	-	4.8	-	-	0.65
Dinoseb	-	11.2	-	-	0.07
MCPA	-	4.1	-	-	249
MCPP	-	3.4	-	-	192

[a] Column conditions are given in Sections 4.1 and 7.5.

TABLE 2.
DETERMINATION OF ESTIMATED QUANTITATION
LIMITS (EQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water (based on one liter sample size)	10
Soil/sediment and other solids	200
Waste samples	100,000

[a]Sample EQLs are highly matrix dependent. The EQLs listed herein are provided for guidance and may not always be achievable.

[b]EQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet weight basis.

TABLE 3.
SINGLE OPERATOR ACCURACY AND PRECISION[a]

Compound	Sample Type	Spike (µg/L)	Mean Recovery (%)	Standard deviation (%)
2,4-D	DW	10.9	75	4
	MW	10.1	77	4
	MW	200	65	5
Dalapon	DW	23.4	66	8
	MW	23.4	96	13
	MW	468	81	9
2,4-DB	DW	10.3	93	3
	MW	10.4	93	3
	MW	208	77	6
Dicamba	DW	1.2	79	7
	MW	1.1	86	9
	MW	22.2	82	6
Dichlorprop	DW	10.7	97	2
	MW	10.7	72	3
	MW	213	100	2
Dinoseb	MW	0.5	86	4
	MW	102	81	3
MCPA	DW	2020	98	4
	MW	2020	73	3
	MW	21400	97	2
MCPP	DW	2080	94	4
	MW	2100	97	3
	MW	20440	95	2
2,4,5-T	DW	1.1	85	6
	MW	1.3	83	4
	MW	25.5	78	5
2,4,5-TP	DW	1.0	88	5
	MW	1.3	88	4
	MW	25.0	72	5

[a]All results based upon seven replicate analyses. Esterification performed using the bubbler method. Data obtained from reference 9.

DW = ASTM Type II
MW = Municipal water

FIGURE 1.
DIAZOMETHANE GENERATOR

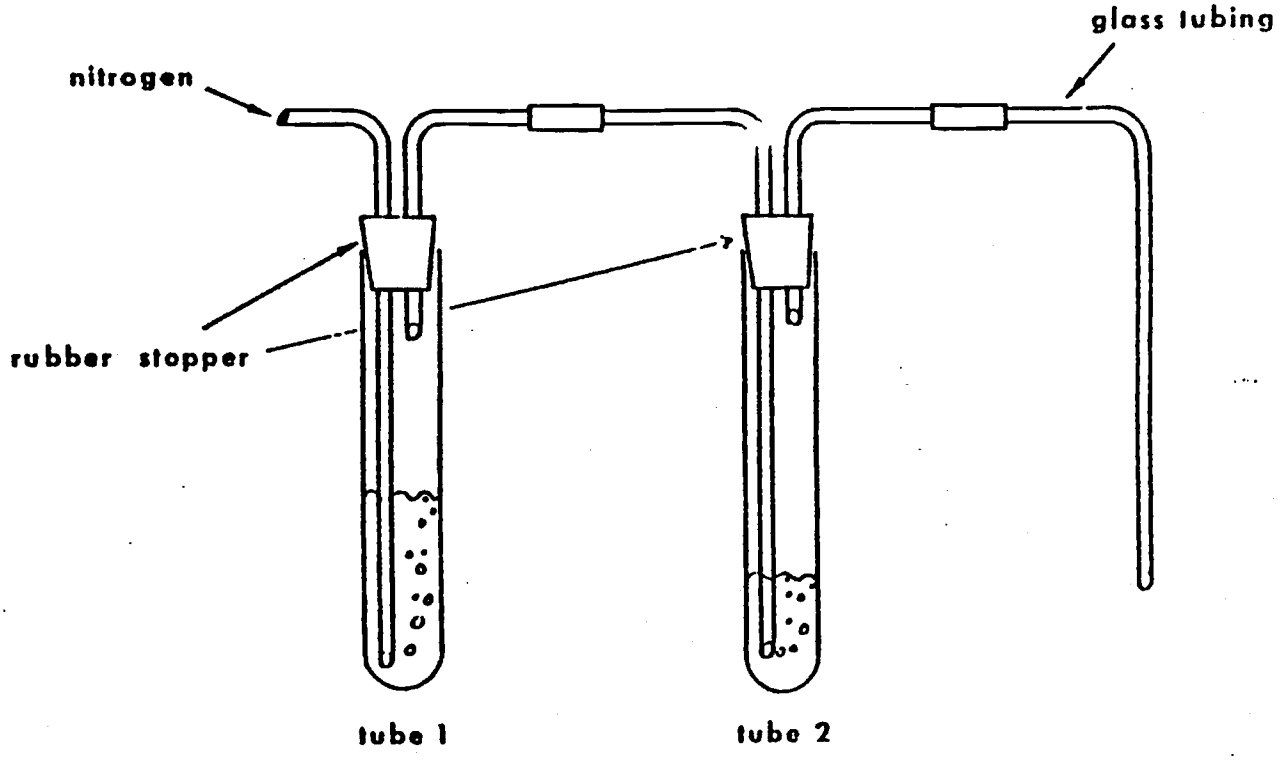


FIGURE 2.
GAS CHROMATOGRAM OF CHLORINATED HERBICIDES

Column: 1.5% SP-2250/1.95% SP-2401 on Supelcoport (100/120 Mesh)
Temperature: Isothermal at 185°C
Detector: Electron Capture

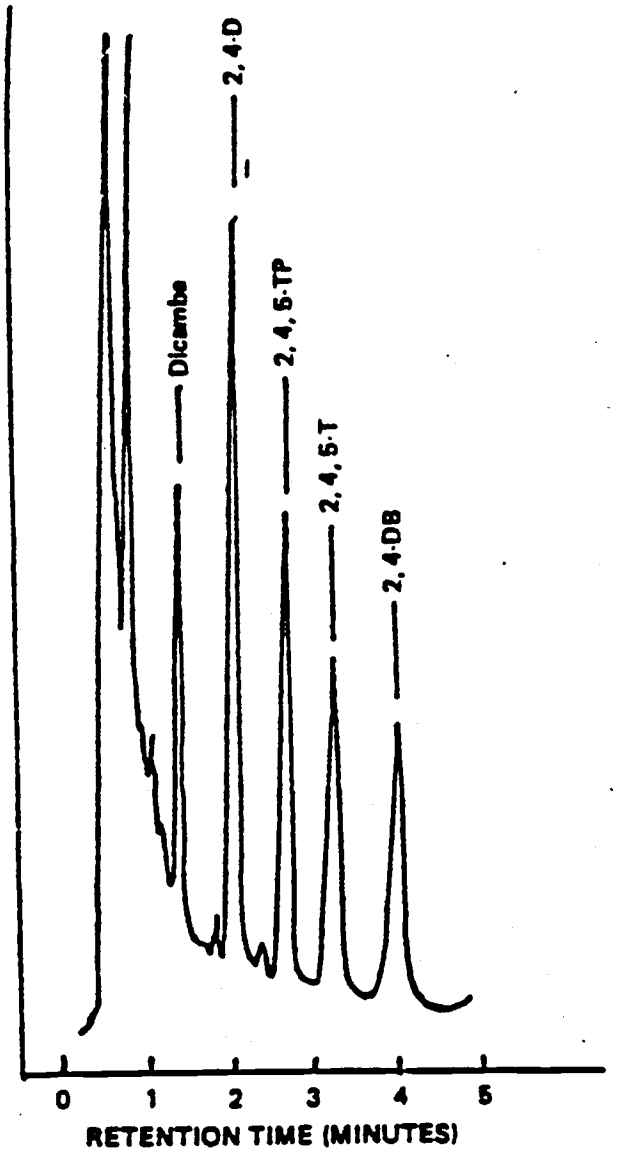


FIGURE 3.
GAS CHROMATOGRAM OF CHLORINATED HERBICIDES

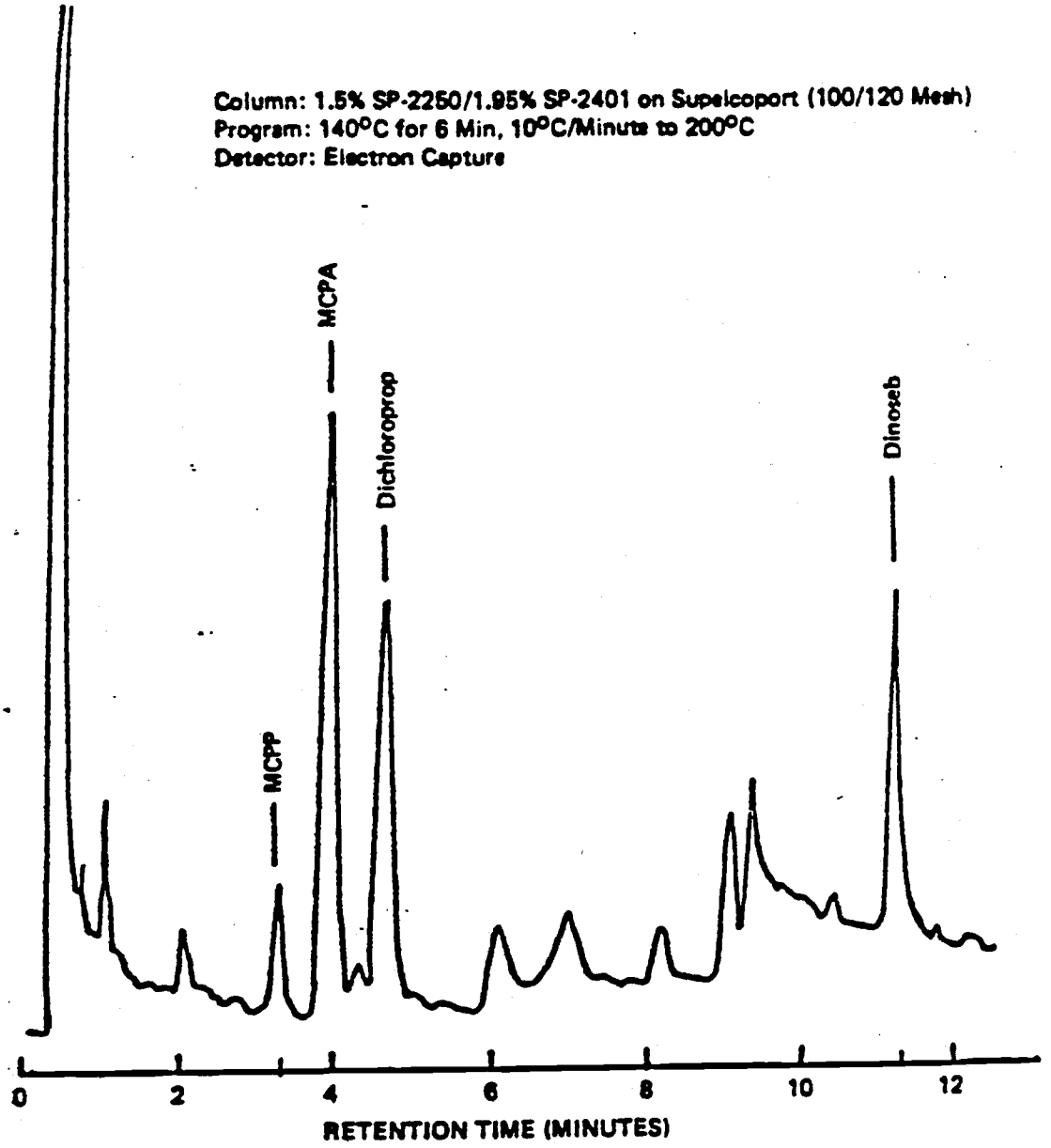
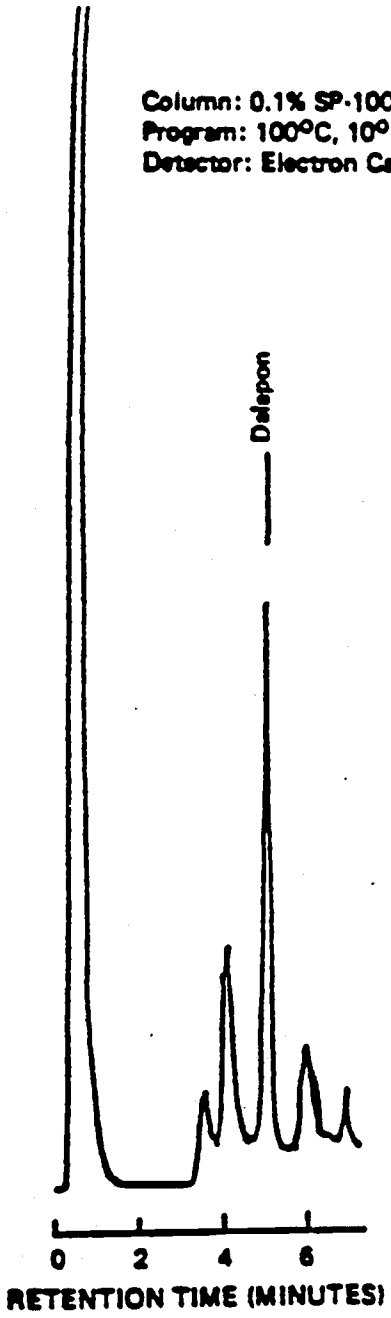


FIGURE 4.
GAS CHROMATOGRAM OF DALAPON, COLUMN 3



CHEMRON INC. - STANDARD OPERATING PROCEDURE 8260

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS
CAPILLARY COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

Standard Operating Procedure (SOP) 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, 0including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. Chemron routinely analyzes for the following analytes:

Analyte	CAS No.[a]
Acetone	67-64-1
Acrolein	107-02-8
Acrylonitrile	107-13-1
Allyl chloride	107-05-1
Benzene	71-43-2
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone (MEK)	78-93-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
2-Chloroethyl vinyl ether	110-75-8
Chloroform	67-66-3
Chloromethane	74-87-3
Dibromochloromethane	124-38-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	141-73-1
1,4-Dichlorobenzene	106-46-7
trans-1,4-Dichloro-2-butene	110-57-6
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
Trans-1,2-Dichloroethene	156-60-5

Analyte	CASE No.[a]
1,2-Dichloropropane	78-87-5
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Diethyl ether	60-29-7
Ethylbenzene	100-41-4
Ethylmethacrylate	97-63-2
2-Hexanone	591-78-6
Iodomethane	74-88-4
Methacrylonitrile	126-98-7
Methylene chloride	75-09-2
Methylmethacrylate	80-62-6
4-Methyl-2-pentanone (MIBK)	108-10-1
Propionitrile	107-12-0
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
Vinyl acetate	108-05-4
Vinyl chloride	75-01-4
o-Xylene	97-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

[a]Chemical Abstract Services Registry Number.

The following compounds can be analyzed using this SOP:

SOP'S 8240/8260 - VOLATILES

Acetone	1,1-Dichloropropene
Acetonitrile	cis-1,3-Dichloropropene
Acrolein (Propenal)	trans-1,3-Dichloropropene
Acrylonitrile	1,2,3,4-Diepoxybutane
Allyl alcohol	1,4-Difluorobenzene
Allyl chloride	1,4-Dioxane
Benzene	Epichlorohydrin
Benzyl chloride	Ethanol
Bromoacetone	Ethylbenzene
Bromobenzene	Ethylene oxide
Bromochloromethane	Ethyl methacrylate
Bromodichloromethane	Hexachlorobutadiene
1-Bromo-4-fluorobenzene	2-Hexanone

Bromoform
Bromomethane
2-Butanone
sec-Butylbenzene
tert-Butylbenzene
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chlorodibromomethane
Chloroethane
2-Chloroethanol
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
Chloroprene
3-Chloropropionitrile
2-Chlorotoluene
4-Chlorotoluene
Dibromochloromethane
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,4-Dichloro-2-butene
dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethene
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
1,2-Dichloropropane
1,3-Dichloropropane
2,2-Dichloropropane
1,3-Dichloro-2-propanol
2-Hydroxypropionitrile
Iodomethane
Isobutyl alcohol
Isopropylbenzene
p-Isopropyltoluene
Malononitrile
Methacrylonitrile
Methylene chloride
Methyl methacrylate
4-Methyl-2-pentanone
Naphthalene
Pentachloroethane
2-Picoline
Propargyl alcohol
b-Propiolactone
Propionitrile
n-Propylamine
n-Propylbenzene
Pyridine
Styrene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
Vinyl acetate
Vinyl chloride
Xylene(s)

1.1 SOP 8260 can be used to quantity most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.2 The practical quantitation limit (PQL) of Method 8260 for an individual compound is approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 3 - 5 µg/L for ground water (5.0 mL sample). PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.3 SOP 8260 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large bore capillary column. The column is temperature programmed to separate the analytes which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph and a jet separator enrichment device.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in solvent to dissolve the volatile organic constituents. A portion of the solution is combined with water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.3 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by an internal standard.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components are avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the purge gas source and the molecular sieve purge gas filter should be checked. Subtracting

blank values from sample results are not permitted. If reporting values not corrected for blanks result in what the laboratory feels is a false positive for a sample, an explaination in text should accompany the uncorrected data.

3.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. Rinse the purging apparatus and sample syringes with two portions of water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high levels of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning. Screening of the samples prior to purge and trap GC/MS analysis can prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique.

3.3 Special precautions are taken to analyze for methylene chloride. The analytical and sample storage area is isolated from the general laboratory sources of methylene chloride to minimize background levels. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing are stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from water and carried through the sampling and handling protocol is as a check on such contamination.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device - The purge-and-trap device consists of two separate pieces of equipment: the Tekmar ALS 2016 multiple sample purger and the Tekmar LSC 2000 containing the trap and the desorber.

4.1.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

4.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The currently approved trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet of this trap to extend its life. Chemron currently uses a VOCARB 3000 (Supelco, Inc., Bellefonte, PA) containing 10.0 cm Carbopack B, 60/80 mesh; 6.0 cm Carboxen 1000, 60/80 mesh; and 1.0 cm Carboxen 1001, 60/80 mesh. Many states approve this trap which offers greater thermal stability to desorb at a higher temperature. Before initial use, the trap is conditioned overnight at the desorption temperature while backflushing with an inert gas flow of at least 20 mL/min, and the trap effluent is vented to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at the desorption temperature while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples. Traps normally last 2-3 months when used daily. Some signs of a deteriorating trap are: uncharacteristic recoveries of surrogates, especially toluene-d_8; a loss of the response of the internal standards during a 12 hour shift; and/or a rinse in the baseline in the early portion of the scan.

4.1.3 The desorber should be capable of rapidly heating the trap to 250°C for desorption. The trap bake-out temperature should not exceed 270°C. These conditions are for the VOCARB 3000. For the Tenax/Silica Gel/Charcoal trap the temperatures are 180 and 225°C respectively.

4.2 Gas chromatograph/mass spectrometer/data system

4.2.1 The currently used Hewlett-Packard 5890 GC is capable of temperature programming and is equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation. A subambient oven controller is available but not used. The GC is interfaced to the MS via a glass lined metal jet separator enrichment device and fused silica tubing.

4.2.2 Gas chromatographic column for Non-cryogenic cooling - 75 m x 0.53 mm i.d. DB-624 wide-bore (J&W Scientific) column with 3 um film thickness. The flow rate of helium carrier gas is established at 8.5 mL/min. The column temperature is held for 8 minutes at 35°C, then programmed to 180°C at 6°C/min, and held for 3.9 minutes or until all expected compounds have eluted. A trap (VOCARB 3000) preheated to 245°C prior to trap desorption is required to provide adequate chromatography of the gas analytes.

4.2.3 Mass spectrometer - Mass spectral data are obtained with electron impact ionization at a nominal electron energy of 70 eV. The current HP 5970 B mass spectrometer meets the requirements of scanning from 35 to 300 amu every 2 seconds or less and produces a mass spectrum that meets all calibration criteria when 50 ng of 4-bromofluorobenzene is introduced into the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

4.2.4 GC/MS interface - The currently used interface consists of a MJSC/HP5890 GC/MS glass lined metal Jet Separator (SGE International, Ringwood, Australia) with a fused silica lined 0.8 mm i.d. transfer tube coupled to a 0.5 mm i.d fused silica transfer line. This arrangement allows greater sensitivity while reducing active sites in the path to the mass spectrometer. This interface gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria. The operating temperature is 150°C - 200°C.

4.2.5 Data system - The current computer system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program and is interfaced to the mass spectrometer. Software allows searching any GC/MS data file for ions of a specified mass, and is capable of plotting an Extracted Ion Current Profile (EICP). This software allows integrating the abundances in any EICP between specified time or scan-number limits. The current system has available a recent version of the NIST/EPA Mass Spectral Library, NIST-EPA MSDC MSDB 1A.

4.3 Moisture Control Module - The MCM minimizes the effect of moisture on analyte response. During the purge mode the MCM is held at 90°C. Before desorbing, the MCM temperature is reduced to 5°C to trap moisture while allowing the analytes to pass through.

4.4 Microsyringes: 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL.

4.5 Syringe valve: Two-way, with Luer ends (three each), if applicable to the purging device.

4.6 Syringe: 5, 10, or 25-mL, gas-tight.

4.7 Balance: Analytical, capable of accurately weighing 0.0001 g, and a Top-loading, capable of accurately weighing 0.01 g.

4.8 Glass scintillation vials: 20-mL, with Teflon lined screw-cap, or glass culture tubes with a Teflon lined screw-cap.

4.9 Vials: 2-mL, crimp seal

4.10 Disposable pipet: Pasteur.

5.0 REAGENTS

5.1 Methanol, CH_3OH: Pesticide quality or equivalent, demonstrated to be free of analytes. Store away from other solvents.

5.2 Nitric acid. Reagent grade.

5.3 ASTM Type II water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified. Must be free of interferents at the method detection limit (MDL) of the analytes of interest.

5.3.1 Currently the water is generated by passing deionized water through a reverse osmosis membrane system with pre and post filters (Ecodyne, Inc., St. Paul, MN). This water is tested for organic contaminants.

5.4 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids or gasses, as appropriate.

5.4.1 Stock standards purchased as prepared solutions are listed in the GC/MS Standards Prep Log for SOP 8260 volatiles.

5.4.2 To prepare a stock standard from pure materials, place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Tare the flask or weigh the flask to the nearest 0.1 mg. Add the assayed reference material, as described below.

5.4.2.1 Liquids - Using a Pasteur pipet, immediately add two or more drops of assayed reference material to the flask; than reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.4.2.2 Gasses - To prepare standards for any compounds that boil below 30°C (e.g. bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.4.2.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter ($\mu g/\mu L$) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Transfer the stock standard solution into a Teflon-sealed screw-cap bottle or a crimped, Teflon-sealed 2 ml vial. Store, with minimal headspace, at -10°C to -20° and protect from light.

5.4.3 Prepare fresh standards for gases every two months or sooner if comparison with check standards indicates a problem Reactive compound such as 2-chloroethylvinyl ether and styrene may need to be prepared more frequently. All other standards must be replaced after six months, or sooner if comparison with check standards indicates a problem. Both gas and liquid standards must be monitored closely by comparison to QC check standards. It may be necessary to replace the standards more frequently if either check exceeds a 25% difference.

5.5 Secondary dilution standards - Using stock standard solution, prepare in methanol, secondary dilutions standards containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace for one week only. The GC/MS Standards Prep Log has a record of working standards preparations.

5.6 Surrogate standards - The current surrogate standards are toluene-d_8, 4-bromofluorobenzene, and 1,2-dichloroethane-d_4. Other compounds may be used as surrogates, depending upon the analysis

requirements. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. Addition of 10 µL of 25 µg/ml of a surrogate standard solution containing each surrogate would be the equivalent of 50.0 µg/L in 5 ml of sample or water.

5.7 Internal standards - The current internal standards are chlorobenzene-d_5, 1,4-difluorobenzene, and bromochloromethane. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Each sample, standard, and blank undergoing GC/MS analysis must be spiked with 10 µL of the internal standard solution prior to analysis. Addition of 10 ul 25 µg/ml of an internal standard solution containing each internal standard would be the equivalent of 50.0 µg/L in 5.0 mL of sample or water.

5.8 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared from pure BFB as decribed in the GC/MS Standards Prep Log.

5.9 Calibration standards - Calibration standards at a minimum of five concentration levels should be prepared from the secondary dilution of stock standards. Prepare these solutions in water. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method. Calibration standards must be prepared daily.

5.10 Matrix spiking standards - Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. At a minimum, the matrix spike for organic volatiles analyses should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. It is desirable to perform a matrix spike using compounds found in samples. Some permits may require spiking specific compounds of interest, especially if they are polar and would not be represented by the above listed compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL for spiking into water samples, and at a concentration of 1000 ug/mL for spiking into 5 mL soil extracts.

5.11 Care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10° to -20°C, preferably in amber bottles with Teflon lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Concentrated waste samples -- Collect in a 125 mL widemouth glass container with Teflon lined lid. No preservative is required. Holding time upon arrival at the laboratory is 14 days.

6.2 Liquid samples -- Collect liquid samples with no residual chlorine present in duplicate 40 mL vials with Teflon lined septum caps. Adjust the pH <2 with H_2SO_4, HCl or solid $NaHSO_4$. Cool and preserve at 4°C. Holding time upon arrival at the laboratory is 14 days.

6.2.1 Liquid samples with residual chlorine present -- Collect in a 125 mL container which has been pre-preserved with 4 drops of 10% sodium thisulfate solution. Gently swirl to mix the sample and transfer to duplicate 40 mL VOA vials with Teflon lined septum caps. Adjust the pH <2 with H_2SO_4, HCl or solid $NaHSO_4$. Cool and preserve at 4°C. Holding time upon arrival at the laboratory is 14 days.

6.3 Soil/Sediments and Sludges -- Collect in a 125 mL widemouth glass container sealed with a septum. Cool and preserve at 4°C. Holding time upon arrival at the laboratory is 14 days.

7.0 PROCEDURE

7.1 Direct injection - In limited applications (e.g. aqueous process wastes) direct injection of the sample into the GC/MS system with a 10 µL syringe may be appropriate. However, the mega bore column is configured for on-column injections, so care is required when injecting the sample. The syringe may break or crack the column end. One such application of direct injection is for verification of the alcohol content of an aqueous sample prior to determining if the sample is ignitable (SOP's 1010 or 1020). In this case, it is suggested that direct injection be used. The detection limit is very high (approximately 10,000 µg/L); it is only permitted when concentrations in excess of 10,000 µg/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection using the same solvent (e.g. water) for standards as the sample matrix (bypassing the purge-and-trap device).

7.2 Initial calibration for purge-and-trap procedure

7.2.1 The GC/MS system must be hardware-tuned to meet the criteria, as described in section 7.3, for a 50 ng purging of 4-bromofluorobenzene (2 µL injection of the BFB standard in 5 ml reagent water). Analyses must not begin until these criteria are met.

7.2.2 A set of at least five calibration standards containing the method analytes is needed. Chemron normally uses a six point calibration curve. One calibration standard should contain each analyte at a concentration approaching but greater than the method detection limit for that compound; the other calibration standards should contain analytes at concentrations that define the range of the method. The routinely used volume of sample for the mega bore capillary column (0.54 mm i.d.) is 5 ml. To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution (e.g. VOA, KTO, CDVA) to an aliquot of water (5 ml) in a gas tight syringe. Use a microsyringe and rapidly inject the alcoholic standard into the gas tight syringe containing the water. Remove the needle as quickly as possible after injection. For each calibration standard add 10 ul each of 25 µg/ml surrogate standard and 25 µg/ml internal standard. Then transfer the contents to a purging device.

7.2.3 Carry out the purge-and-trap procedure as described in Section 7.4.1.

7.2.4 Tabulate the area response of the characteristic quantitation ion of a compound against the concentration of that compound for each analyte, internal standard and surrogate standard in the calibration curve. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = \frac{(A_x \times C_{is})}{(A_{is} C_x)}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

7.2.5 The average RF must be calculated for each compound and recorded for archiving. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2-

tetrachloroethane, and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.2.5.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

7.2.5.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.2.5.3 Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.6 Using the RFs form the initial calibration, calculate the percent relative standard deviation (%RSD)) for Calibration Check Compounds (CCCs). Record the %RSDs for all compounds for archiving. The percent RSD is calculated as follows:

$$\%RSD = \frac{SD}{x} \times 100$$

Where:

RSD = Relative standard deviation.
RF = RF for an single level
x = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

$$SD = \sqrt{\frac{(\sum ((RF-x)^2))}{N-1}}$$

The %RSD for each individual CCC must be less than 30 percent. This criterion must be met for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene, chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl chloride.

If the CCCs are not required analytes by the permit, then all required analytes must meet the 30% RSD criterion.

7.3 Daily GC/MS calibration

7.3.1 Prior to the analysis of samples, purge 50-ng of 4-bromofluorobenzene. Certain criteria must be met each 12-hour period from the resultant mass spectra for the BFB. The following are the 4-BFB criteria:

Target Mass	Comparison Mass	Lower Limit,%	Upper Limit,%
50	95	15	40
75	95	30	60
95	95	100	100
96	95	5	9
173	174	0	2
174	95	50	100
175	174	5	9
176	174	95	101
177	176	5	9

7.3.2 The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC (Step 7.3.3) and CCC (Step 7.3.4).

7.3.3 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.3.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed in section 7.2.6 are used to check the validity of the initial calibration. Calculate the percent difference using the following equation:

$$\% \ Difference = \frac{(RF_I - RF_c)}{RF_I} \times 100$$

Where:

RF_I = Average response factor from initial calibration.
RF_c = Response factor from daily midpoint standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (>25% difference), for any one CCC, corrective action must be taken. If the source of the problem cannot be determined after corrective action has been taken, a new calibration curve must be generated. This criterion must be met before quantitative sample analysis begins. If the CCCs are not required analytes by the permit, then all required analytes must meet the 25% difference criterion.

7.3.5 The internal standard responses and retention times of the daily check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.4 GC/MS analysis

7.4.1 Water samples

7.4.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system.

7.4.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.4.1.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Step 7.3) before analyzing samples.

7.4.1.4 The purge gas (helium) flow rate on the purge-and-trap device was determined as optimized at 28-32 mL/min.

7.4.1.5 To prepare a sample for analysis, open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully draw out a portion of the sample with the 5 mL syringe, or 25 mL syringe if lower detection limits are required, and rinse the syringe once with the sample. Draw out greater than 5 mL of sample (or 25 mL), and compress the sample with the syringe plunger to vent any residual air. After the air is vented, adjust the sample volume to 5.0 mL (or 25 mL). This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 25-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.4.1.6 Purgeable samples may require dilution.

7.4.1.7 When compositing samples prior to GC/MS analysis, the samples must be cooled at 4°C to minimize volatilization losses.

7.4.1.8 Add 10.0 µL of surrogate spiking solution and 10.0 µL of internal standard spiking solution through the end bore of the syringe. The addition of 10 µL of the surrogate spiking solution to 5 mL of sample is equivalent to a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the internal standard spiking solution to 5 mL of sample is equivalent to a concentration of 50 µg/L of each internal standard.

7.4.1.9 Attach the syringe-syringe valve assembly to the syringe valve of one of the auto sampler positions of the Tekmar ALS 2016 purging device. Open the syringe valve and inject the sample into the purging chamber.

7.4.1.10 Close the valve and purge the sample for 11.0 ± 0.1 minutes at ambient temperature. Be sure the trap is cooler than 32°C.

7.4.1.11 Sample desorption - The mode of sample desorption is determined by the type of capillary column employed for the analysis. When using a mega bore capillary column an all glass jet separator must be interfaced to the MS.

7.4.1.11.1 After the 11 minute purge and a 3 minute dry purge, the moisture control module should cool to 5°C, and the sample is ready to desorb. After all preset temperatures are reached, the purge and trap system moves to desorb pre-heat, then to the desorb mode. Simultaneously, the temperature program sequence of the gas chromatograph is initiated and data acquisition is started. The trapped materials are introduced to the GC column by rapidly preheating the trap (VOCARB 3000) to 245°C, then heating to 250°C for desorption while backflushing the trap with an inert gas at 8-15 mL/min for 4 minutes. While the purged analytes are being introduced into the gas chromatograph, the autosampler drains the purging vessel. Using the sample syringe, wash the purge glassware chamber with with two 5 (or 25 mL) portions of water.

7.4.1.11.2 The column temperature is programmed to start at 35°C for 10 minutes, then ramped at 6°C/min to 180°C and held for 5.8 minutes or until all the analytes elute.

7.4.1.11.3 After desorbing the sample for 4 minutes, the trap is reconditioned in the bake mode. The trap temperature rises to 270°C and held for 10 minutes. When the trap cools, the next sample can be analyzed.

7.4.1.12 If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.4.1.15 For matrix spike analysis, add 10 µL of the matrix spike solution to 5 mL of sample. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.4.1.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.4.2 Water-miscible liquids

7.4.2.1 Water-miscible liquids are analyzed as water samples. Initially screening of diluted samples are recommended for any sample suspected of containing analytes which may overload and contaminate the column.

7.4.2.2 Initial and serial dilutions can be prepared by pipeting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with water. Transfer immediately to a 5-mL gas-tight syringe.

7.4.2.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with water by adding at least 20 µL, but not more than 100-µL of liquid sample. The sample is ready for addition of internal and surrogate standards.

7.4.3 Sediment/soil and waste samples - It is recommended that all samples of this type be screened prior to the purge-and-trap GC/MS analysis.

7.4.3.1 Low-level method - This is designed for samples containing individual purgeable compounds of <1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). Weigh a 5 g sample into a sparge vessel if the expected concentration is <0.1 mg/kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/kg. The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Mix the soil and 5 ml of water containing 10 µL each of the surrogate and internal standards. Analyze all blanks and standards under the same conditions as the samples.

7.4.3.1.1 Determine the percent moisture of the soil/sediment sample. This includes waste samples that are amenable to moisture determination. Other wastes should be reported on a wet-weight basis. Immediately after weighing the sample, weigh (to 0.1 g) 5-10 g of additional sediment/soil into a tared crucible. Dry the contents of he crucibles overnight at 105°C. Allow to cool in a desiccator and reweigh the dried contents. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\% \textit{ moisture} = \frac{\textit{grams of sample - grams of dry sample}}{\textit{grams of sample}} \times 100$$

7.4.3.1.2 Matrix spike for low-level sediment/soils: Add 10 µL of the VMS 25 matrix spike solution to the 5 mL of water. The concentration for a 5 g sample would be equivalent to 50 ug/kg of each matrix spike standard.

7.4.3.2 High-level method - The method is based on extracting the sediment/soil with methanol. For sediment/soil wastes that are soluble in methanol weigh 6 g (wet weight) of sample into a tared culture tube. The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Pipet 5.0 mL of methanol into the vial and cap the tube. If the sample is one of the duplicate matrix spike spike samples, add the spike before extraction. Using a cyclomixer, extract the soil, then centrifuge to separate the soil from the extract. Take 50 µL of the extract and add it to 5.0 mL of water containing 10 µL each of surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/kg should be analyzed by this method.

7.4.3.2.1 Matrix spike for high-level sediment/soil samples: Add 25 µL VMS 1000 to the culture tube containg sample and 5 mL of methanol. Add a 50 µL aliquot of this extract to 5 mL of water for purging.

7.5 Data interpretation

7.5.1 Qualitative analysis

7.5.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference should be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

7.5.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.5.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100% must be present in the sample spectrum). (2) The relative intensities of ions specified in (1) must agree within ± 20% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.

7.5.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should e reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

7.5.2 Quantitative analysis

7.5.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.5.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste:

$$concentration\ (\mu g/L) = \frac{(A_x)\ (I_s)}{(A_{is})\ (RF)\ (V_o)}$$

Where:

A_x = Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured (Step 7.2.6).
V_o = Volume of water purged (mL), taking into consideration any dilutions made.

Sediment/Soil, Sludge, and Waste:

High-level:

$$concentration\ (\mu g/kg) = \frac{(A_x)\ (I_s)\ (V_t)}{(A_{is})\ (RF)\ (V_i)\ (W_s)}$$

Low-level:

$$concentration\ (\mu g/kg) = \frac{(A_x)\ (I_s)}{(A_{is})\ (RF)\ (W_s)}$$

Where:

A_x, I_s, A_{is}, RF	=	Same as in water and water-miscble waste above.
V_t	=	Volume of total extract (μL) (use 10,000 μL or a factor of this when dilutions are made).
V_i	=	Volume of extract added (μL) for purging.
W_s	=	Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

7.5.2.3 Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The %moisture of the sample should be reported as specified.

7.5.2.4 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulas given above should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.5.2.5 Report results without correction for recovery data. When duplicates and spiked samples are analyzed, report all data obtained with the sample results.

8.0 QUALITY CONTROL

8.1 A formal quality control program is required. The minimum requirements of this program consist of an initial demonstration of laboratory capability (see GC/MS Analytical Parameters binder) and an ongoing analysis of spiked samples to evaluate and document quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in a in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a calibration blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank should be processed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.3 Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

8.4 Required instrument QC

8.4.1 The GC/MS system must be tuned to meet the BFB specifications in Section 7.3.1.

8.4.2 There must be an initial calibration of the GC/MS system as specified in Section 7.2.2.

8.4.3 The GC/MS system must meet the SPCC criteria specified in Step 7.3.3 and the CCC criteria in Step 7.3.4, each 12 hours.

8.5 To establish the ability to generate acceptable accuracy and precision on water samples, the analyst must perform the following operations.

8.5.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 2.0 µg/mL in methanol. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate must be made using stock standards prepared independently form those used for calibration.

8.5.2 Prepare a QC reference sample to contain 20 µg/L of each analyte by adding 5.0 µL of QC reference sample concentrate (VOAQC) to 5 mL of water.

8.5.3 Four 5 mL aliquots of the well-mixed QC reference sample are analyzed according to the method beginning in Section 7.4.1.

8.5.4 Calculate the average percent recovery (R) and the standard deviation of the percent recovery (S_R), for the results. Ground water background corrections must be made before R and S_R calculation.

8.5.5 Single laboratory recovery and precision data obtained for the method analytes from water are listed at the end of this document. Results are comparable if the calculated percent relative standard deviation (RSD) does not exceed 2.6 times the single laboratory RSD or 20%, whichever is greater and the mean recovery lies within the interval R ± 3S or R ± 30%, whichever is greater.

8.6 The volatiles analyst must, on an ongoing basis, analyze a blank and spiked replicates for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of spiked replicates. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.6.1 The concentration of the spike in the sample should be determined as follows:

8.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.6.2, whichever concentration would be larger.

8.6.1.2 If the concentration of a specific analyte in a water sample is not being checked against a specific limit, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Step 8.6.2, whichever concentration would be larger. For other matrices, the recommended spiking concentration is 10 times the PQL.

8.6.2 Analyze one 5 mL sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC check sample concentrate (Section 8.5.1) appropriate for the background concentration in the sample. Spike a second 5 mL sample aliquot with the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as 100(A-B)%/T, where T is the known true value of the spike.

8.6.2.1 Compare the percent recovery (R_i) for each analyte with QC acceptance criteria established from the analyses of laboratory control standards.

8.6.2.2 If recovery is not within limits, the following procedures are required.

8.6.2.2.1 Check to be sure there are no errors in calculations, matrix spike solutions and internal standards. Also, check instrument performance.

8.6.2.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

8.6.2.2.3 If the checks in 8.6.2.2.1 reveal no errors, the recovery problem encountered with the spiked sample is judged to be matrix-related, non system-related. The result for that analyte in the unspiked sample is labeled suspect/matrix to inform the user that the results are suspect due to matrix effects.

8.7 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix) as in Section 8.6, calculate the average percent recovery (p) and the standard deviation of the percent recovery (Sp). Express the accuracy assessment as a percent recovery interval from p - $2S_p$ to p + $2S_p$. If p = 90% and S_p = 10%, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis.

8.8 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed.

8.8.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

CHEMRON INC. - STANDARD OPERATING PROCEDURE 8270

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR SEMIVOLATILE ORGANIC: CAPILLARY COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications.

1.2 SOP 8270 is used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicon. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Under the alkaline conditions of the extraction step, a-GHC, 7-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction is performed if these compounds are expected. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4 The practical quantitation limit (PQL) of SOP 8270 for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 2). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 Chemron restricts the use of this method to analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst demonstrates the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples are prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and for their qualitative and quantitative analysis by mass spectrometry.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes are evaluated for interferences. If inteferences are present and their source is determined to be in the preparation and/or cleanup of the samples, corrective action is taken to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it is followed by the analysis of solvent to check for cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system:

4.1.1 Gas chromatograph: Chemron is equipped with an analytical system complete with the Hewlett-Packard Model 5890 Series II GC, a temperature-programmable gas chromatograph suitable for splitless injection, and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

4.1.2 Column: 25-m x 0.20-mm I.D. .33-μm film thickness silicon-coated fused-silica capillary column (J&W Scientific DB-5ms PN 128-5522).

4.1.3 Mass spectrometer: Chemron utilizes the Hewlett-Packard Model No. 5970 B Mass Spectrometer, capable of scanning from 35 to 500 amμ every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The HP 5970 B is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 3 when 1 μL of the GC/MS tuning standard is injected through the GC (50 ng per injection for each compound of interest) and achieves acceptable tuning performance criteria.

4.1.4 GC/MS interface: Direct capillary coupling of GC-to-MS gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

4.1.5 Data system: A CompuAdd 486 DX PC system is interfaced to the HP 5970 B mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer operates on HP DOS Chemstation software (Ver. B.00.01) that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software also allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is also installed into the system.

4.2 Syringe: 10 μL.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground glass stoppers.

4.4 Balance - Analytical, 0.0001 g

4.5 Bottles - Glass with Teflon-lined crimp tops.

5.0 REAGENTS

5.1 Stock standard solutions (2.00 μg/μL): Standard solutions are prepared from pure standard materials and/or purchased as certified solutions from Ultra Scientific, RI.

5.1.1 Stock standard solutions are prepared by accurately weighing about 0.2500 g of pure material, dissolving the material in pesticide quality acetone, and diluting to volume in a 25-mL volumetric flask. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards certified by the manufacturer are also used at 2.0 μg/μL concentration.

5.1.2 The stock standard solutions are transferred into Teflon-sealed screw-cap bottles. The bottles are then stored at 4°C and protected from light. Stock standard solutions are checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.1.3 Stock standard solutions are replaced after one year or sooner if comparison with quality control check samples indicates a problem.

5.2 Internal standard solutions: The internal standards being used are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. A commercially prepared mixture from Ultra Scientific, RI is being used as a certified solution. The purchased solution contains each standard at a concentration of 4,000 ng/μL. Each 1-mL sample extract undergoing analysis is spiked with 10 μL of the internal standard solution, resulting in a concentration of 40 ng/μL of each internal standard. Solutions are stored at 4°C or less when not being used.

5.3 GC/MS tuning standard: A methylene chloride solution containing 50 ng/μL of decafluorotriphenylphosphine (DFTPP) is prepared. The standard also contains 50 ng/μL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Solutions are stored at 4°C or less when not being used.

5.4 Calibration standards - Calibration standards at a minimum of five concentration levels are prepared. One of the calibration standards is at a concentration near, but above, the method detection limit; The others correspond to the expected range of concentrations found in real samples but do not exceed the working range of the GC/MS system. Each standard contains each analyte for detection by this SOP (e.g. some or all of the compounds listed in Table 1 may be included). Each 1-mL aliquot of calibration standard is spiked with 10 μL of the internal standard solution prior to analysis. All standards are stored at 4°C or less and are freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard is prepared weekly and stored at 4°C.

5.5 Surrogate standards - The surrogate standards used are phenol-d_5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluoro-biphenyl, and p-terphenyl-d_{14}. See SOP 3500 for the explanation of surrogate standards preparation. The analyst determines what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. This concentration is injected into the GC/MS to determine recovery of surrogate standards in all blanks, spikes, and sample extracts. All dilutions of sample extracts are taken into account.

5.6 Matrix spike standards: See SOP 3500 for explanation of matrix spike standard preparation. The analyst determines what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. This concentration is injected into the GC/MS to determine recovery of surrogate standards in blanks, spikes, and sample extracts. All dilutions of sample extracts are taken into account.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Chemron's sampling, preservation, and handling techniques comply with the requirements stated in SW-846 in the chapter, Organic Analytes, Step 4.1.

7.0 PROCEDURE

7.1 Sample preparation: Samples are prepared by one of the following methods prior to GC/MS analysis.

Matrix	Chemron SOP
Water	3510, 3520
Soil/sediment	3540, 3550
Waste	3540, 3550, 3580

7.1.1 Direct injection - In very limited applications direct injection of the sample into the GC/MS system with a 10 µL syringe may be appropriate. The detection limit is very high (approximately 10,000 µg/L); therefore, it is only performed when concentrations in excess of 10,000 µg/L are expected. The system is calibrated by direct injection.

7.2 Extract cleanup: Extracts are cleaned up by any of the following methods prior to GC/MS analysis.

Compounds	Chemron SOP
Phenols	3630, 3640
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3620, 3640, 3660
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620, 3640
Petroleum waste	3611, 3650
All priority pollutant base, neutral, and acids	3640

7.3 Initial calibration - The GC/MS operating conditions:

Mass Range: 35-550 amμ
Scan time: .5 sec/scan
Initial column temperature and hold time: 40° for 5 min
Column temperature program: 40-290°C at 10°/min
Final column temperature : 290°C for 6 min. then increases 20°/min. to 325°C and maintains for 10 min.
Injector temperature: 250°C
Transfer line temperature: 325°C
Injector: Grob-type, splitless
Sample volume: 1-2 μL
Carrier gas: Helium at 34 cm/sec.

7.3.1 The GC/MS system is hardware-tuned to meet the criteria in Table 3 for a 50 ng injection of DFTPP. Analyses do not begin until all these criteria are met. Background subtraction is straightforward and designed only to eliminate column bleed or instrument background ions. The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. If degradation is excessive and/or poor chromatography is noted, the injection port may be cleaned. It may also be necessary to break off the first 6-12 in. of the capillary column.

7.3.2 The internal standards selected in Paragraph 5.1 permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. The base peak ion from the specific internal standard is used as the primary ion for quantitation (see Table 1). If interferences are noted, the next most intense ion is used as the quantitation ion, i.e., for 1,4-dichlorobenzene-d_4 m/z 152 is used for quantitation.

7.3.3 1 μL of each calibration standard is analyzed (containing internal standards) and the area of the primary characteristic ion is tabulated against concentration for each compound (as indicated in Table 1). Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes. Response factors (RFs) are calculated for each compound as follows:

$$RF = (A_x) / (A_{is}C_x)$$

Where:

A_x = Area of characteristic ion for the compound being measured.
A_{is} = Area of characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the compound being measured.

7.3.4 The average RF is calculated for each compound. The percent relative standard deviation (%RSD = 100[SD/RF]) is also calculated for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (See Table 4) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreements.

7.3.5 A system performance check is performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds (SPCCs) is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.4 Daily GC/MS calibration:

7.4.1 Prior to the analysis of samples, the GC/MS tuning standard is analyzed. A 50-ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 3. These criteria are demonstrated during each 12-hr shift.

7.4.2 A calibration standard(s) at 50 µg/mL concentration containing all semivolatile analytes, including all required surrogates, is performed every 12-hr during analysis. The response factor data from the standards are compared every twelve hours with the average response factor from the initial calibration for a specific instrument as per the SPCC (Paragraph 7.4.3) and CCC (Paragraph 7.4.4) criteria.

7.4.3 System Performance Check Compounds (SPCCs) - A system performance check is made every 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system is evaluated, and corrective action is taken before sample analysis begins. The minimum response factor for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

7.4.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed in Table 4 are used to check the validity of the initial calibration. The percent difference is calculated using the following equation:

$$\% \ Difference = \frac{RF_I - RF_c}{RF_I} \times 100$$

Where:

RF_I = Average response factor from initial calibration.
RF_c = Response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory considers this a warning limit. If the percent difference for each CCC is less than 30%, the initial calibration is assumed to be valid. If the criterion is not met (>30% difference), for any one CCC, corrective action is taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration is generated. This criterion MUST be met before quantitative sample analysis begins.

7.4.5 The internal standard responses and retention times in the check calibration standard are evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system is inspected for malfunctions and corrections are made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer is inspected for malfunctions and corrections are made, as appropriate.

7.5 GC/MS analysis:

7.5.1 The extract may be screened on a GC/FID or GC/PID using the same type of capillary column in order to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 The 1-mL extract obtained from sample preparation is spiked with 10 µL of the internal standard solution just prior to analysis.

7.5.3 The 1-mL extract is analyzed by GC/MS using a 25-m x 0.20-mm DB-5ms capillary column. The volume to be injected should ideally contain 100 ng of base/neutral and 200 ng of acid surrogates (for a 1 µL injection). The GC/MS operating conditions used are specified in Paragraph 7.3.

7.5.4 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution takes place. Additional internal standards are added to the diluted extract to maintain the required 40 ng/µL of each internal standard in the extracted volume. The diluted extract is reanalyzed.

7.5.5 All qualitative and quantitative measurements are performed as described in Paragraph 7.6. The extracts are stored at 4°C, protected from light in crimp-top vials equipped with unpierced Teflon-lined septa.

7.6 Data interpretation:

7.6.1 Qualitative analysis:

7.6.1.1 An analyte (e.g. those listed in Table 1) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference is obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference are obtained through analysis of the calibration standards. Two criteria must be satisfied to

verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

7.6.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard is run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT is assigned by using extracted ion current profiles for ions unique to the component of interest.

7.6.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100% must be present in the sample spectrum).

7.6.1.1.3 The relative intensities of ions specified in Paragraph 7.6.1.1.2 must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.

7.6.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.6.2 Quantitative analysis:

7.6.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification takes place using the internal standard technique. The internal standard used is the one nearest the retention time of that of a given analyte (e.g. see Table 5).

7.6.2.2 The concentration of each identified analyte in the sample is calculated as follows:

Water:

$$concentration\ (\mu/L) = \frac{(A_x)\ (I_s)\ (V_t)}{(A_{is})\ (RF)\ (V_o)\ (V_i)}$$

Where:

A_x = Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
V_t = Volume of total extract, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean V_t = 10,000 μL. If half the base/neutral extract and half the acid extract are combined, V_t = 2,000.
A_{is} = Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured (Step 7.3.3).
V_o = Volume of water extracted or diluted in grams.
V_i = Volume of extract injected (μL).

Sediment/Soil Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

$$concentration\ (\mu g/kg) = \frac{(A_x)\ (I_s)\ (V_t)}{(A_{is})\ (RF)\ (V_i)\ (W_s)\ (D)}$$

Where:

A_x, I_s, A_{is}, V_t, RF, V_i = Same as for water.
W_s = Weight of sample extracted or diluted in grams.
D = (100 - % moisture in sample)/100, or 1 for a wet-weight basis.

7.6.2.3 Where applicable, an estimate of concentration for noncalibrated components in the sample is made. The formulas given above are used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained is reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. The nearest internal standard free of interferences is used.

7.6.2.4 Results are reported without correction for recovery data. When duplicates and spiked samples are analyzed, all data obtained with the sample results are reported.

7.6.2.5 Quantitation of multicomponent compounds (e.g., Aroclors) is beyond the scope of SOP 8270. Normally, quantitation is performed using a GC/ECD by SOP 8080.

8.0 QUALITY CONTROL

8.1 At Chemron, a formal quality control program is in place in order to ensure proper adherence to all quality control requirements. This program consists of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard is analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst demonstrates, through the analysis of a calibration blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank is processed as a safeguard against chronic laboratory contamination. The blanks are carried through all stages of sample preparation and measurement steps.

8.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard is evaluated to determine if the chromatographic system is operating properly. Questions that are asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), the system is r e c a l i b r a t e d

8.4 Required instrument QC is found in the following sections:

8.4.1 The GC/MS system is tuned to meet the DFTPP specifications in Section 7.3.1 and 7.4.1.

8.4.2 There is an initial calibration of the GC/MS system as specified in Step 7.3.

8.4.3 The GC/MS system must meet the SPCC criteria specified in Step 7.4.3 and the CCC criteria in Step 7.4.4, every 12 hours.

8.5 To establish the ability to generate acceptable accuracy and precision on water samples, the analyst performs the following operations.

8.5.1 A quality control (QC) reference sample concentrate containing each analyte at a concentration of 10 µg/mL in methanol is analyzed. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate is made using stock standards prepared independently from those used for calibration.

8.5.2 Using a pipet, QC check samples are prepared at a concentration of 100 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.5.3 The well-mixed QC check samples are analyzed according to the method beginning in Section 7.1 with extraction of the samples.

8.5.4 The average percent recovery (X) in µg/L, and the standard deviation of the recovery (s) in µg/L, are calculated for each analyte of interest using the four results.

8.5.5 For each analyte, s and x are compared with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and x exceed the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are analyzed.

8.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst proceeds according to Step 8.5.6.1 or 8.5.6.2.

8.5.6.1 The source of the problem is located and corrected and the test is repeated for all analytes beginning with Step 8.5.2.

8.5.6.2 Beginning with Step 8.5.2, the test is repeated only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, the source of the problem is located and corrected and the test is repeated for all compounds of interest beginning with Step 8.5.2.

8.6 Chemron analyzes, on an ongoing basis, a blank and spiked replicates for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of spiked replicates. When analyzing only one to ten samples per month, at least one spiked sample per month is performed.

8.6.1 The concentration of the spike in the sample is determined as follows:

8.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Step 8.6.2, whichever concentration would be larger.

8.6.1.2 If the concentration of a specific analyte in the sample is not being checked against a specific limit, to the analyte, the spike should be at 100 ug/L or 1 to 5 times higher than the background concentration determined in Section 8.6.2, whichever concentration would be larger.

8.6.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be at (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 100 µg/L.

8.6.2 One sample aliquot is analyzed to determine the background concentration (B) of each analyte. If necessary, a new QC check sample concentrate is prepared (Step 8.5.1) appropriate for the background concentration in the sample. A second sample aliquot is spiked with 1.0 mL of the QC check sample concentrate and analyzed in order to determine the concentration after spiking (A) of each analyte. Each percent recovery (p) is calculated as 100(A-B)%/T, where T is the known true value of the spike.

8.6.3 The percent recovery (p) for each analyte is compared with QC criteria found in Table 6. These acceptance criteria were calculated to include an allowance for error in measurement on both the background and spike concentrations, assuming a spike to background ration of 5:1. This error will be accounted for to the extent that the analyst's spike to background ration approaches 5:1. If spiking was performed at a concentration lower than 100 ng/L, the analyst must use either the QC acceptance criteria presented in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration.

8.6.3.1 In addition to the EPA lab-established control limits listed in the QC Acceptance Criteria in Table 6, Chemron has calculated, and regularly updates, it's own in-lab-established control limits for the various spiked compounds in the various matrices. The calculation is performed as follows:

Upper Control Limit (UCL) = p + 3S

Lower Control Limit (LCL) = p - 3S

8.6.4 If any individual p falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria is analyzed as described in Section 8.7.

8.7 If any analyte fails the acceptance criteria for recovery in Section 8.6, a QC check standard containing each analyte that failed is prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of analytes being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of analytes in Table 6 must be measured in the sample in Section 8.6, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard is routinely analyzed with the spiked sample.

8.7.1 The QC check standard is prepared by adding 1.0 mL of the QC check sample concentrate (Section 8.5.1 or 8.6.2) to 1 L of reagent water. The QC check standard needs only to contain the analytes that failed criteria in the test in Section 8.6.

8.7.2 The QC check standard is analyzed to determine the concentration measured (A) of each analyte. Each percent recovery (p_s) is calculated as 100 (A/T)%, where T is the true value of the standard concentration.

8.7.3 The percent recovery (p_s) for each analyte is compared with the corresponding QC acceptance criteria found in Table 6. Only analytes that failed the test in Section 8.6 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem is immediately identified and corrected. The analytical result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.8 As part of our QC program, Chemron assesses and documents method accuracy for each matrix studied. After the analysis of five spiked samples of the same matrix as in Step 8.6, the average percent recovery (p) and the standard deviation of the percent recovery (Sp) are calculated. The accuracy assessment is expressed as a percent recovery interval from $p - 2S_p$ to $p + 2S_p$. The accuracy assessment is updated for each analyte on a regular basis.

8.9 To determine acceptable accuracy and precision limits for surrogate standards the following procedures are performed.

8.9.1 For each sample analyzed, the percent recovery of each surrogate in the sample is calculated.

8.9.2 Once a minimum of thirty samples of the same matrix have been analyzed, the average percent recovery (p) and standard deviation of the percent recovery (s) is calculated for each of the surrogates.

8.9.3 For a given matrix, the upper and lower control limit for method performance for each surrogate standard has been calculated as follows:

Upper Control Limit (UCL) = p + 3S
Lower Control Limit (LCL) = p - 3S

8.9.4 For aqueous and soil matrices, these in-lab established surrogate control limits are, if applicable, compared with the control limits listed in Table 8. The limits given in Table 8 are multi-laboratory performance based limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Step 8.9.3 must fall within those given in Table 8 for these matrices. Therefore, if the upper or lower control limit generated by Chemron does not fall within the range specified in Table 8, the upper or lower control limit listed in Table 8 is adopted as the control limit value listed in Table 9.

8.9.5 If recovery is not within these limits, the following procedures are performed.

1) Calculations, surrogate solutions and internal standards are checked to ensure their accuracy. Also, instrument performance is checked.

2) The data is recalculated and/or the extract is reanalyzed if any of the above checks reveal a problem.

3) If none of the above are a problem, the sample is reextracted and reanalyzed or the data is flagged as "estimated concentration."

8.9.6 At a minimum, Chemron updates recovery limits on a matrix-by-matrix basis, bi-monthly.

8.10 Chemron has adopted additional quality assurance practices for use with this method. When doubt exists over the identification of the peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column or a different ionization mode using a mass spectrometer is used. Whenever possible, the standard reference materials are analyzed. Chemron regularly generates and maintains control charts that track analyst performance and present recovery and precision data in graph and tabular format. Also, Chemron regularly participates in relevant performance evaluation studies.

Bibliography

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Method 625, "October 26, 1984.
2. U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, July 1985, Revision.
3. Provost, L.P.; Elder R.S. "Interpretation of Percent Recovery Data"; American Laboratory 1983, 15, 58-63.
4. Eichelberger, J.W., L.E. Harris, and W.L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry," Analytical Chemistry, 47, 995, 1975.
5. "Method Detection Limit for Methods 624 and 625," Olynyk, P., W.L. Budde, and J.W. Eichelberger, Unpublished report, October 1980.
6. "Interlaboratory Method Study for EPA Method 625-Base/Neutrals, Acids, and Pesticides," Final Report for EPA Contract 68-03- 3102 (in preparation).
7. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48. 1037, 1965.

Rev. 2.0

Date - December 6, 1994

Approved: _____ Date: 12/06/94

TABLE 1.
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75[a]	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d_4 (I.S.)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,88,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	62,44,45,74
Thiophenol (Benzenethiol)	7.42	110	110,66,109,84
Methyl methanesulfonate	7.48	80	80,79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	54,98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	102,42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,108,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	110,79,95,109,140
Ethyl methanesulfonate	9.62	79	79,109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d_8 (I.S.)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	99,155,127,81,109
Diethyl sulfate	11.37	139	139,45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	107,108,77,79,90
Hexachloropropene	12.45	213	213,211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272
N-Nitrosopyrrolidine	12.65	100	100,41,42,68,69
Acetophenone	12.67	105	71,105,51,120
4-Methylphenol	12.82	107	107,108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	106,107,77,51,79
3-Methylphenol	12.93	107	107,108,77,79,90
2-Chloronaphthalene	13.30	162	127,164

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
N-Nitrosopiperidine	13.55	114	42,114,55,56,41
1,4-Phenylenediamine	13.62	108	108,80,53,54,52
1-Chloronaphthalene	13.65[a]	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	106,141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	104,76,50,148
o-Anisidine	15.00	108	80,108,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d_{10} (I.S.)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	162,164,126,98,63
4-Chloroaniline	15.50	127	127,129,65,92
Isosafrole	15.60	162	162,131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	121,122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00[a]	143	115,116
1,4-Naphthoquinone	16.23	158	158,104,102,76,50,130
p-Cresidine	16.45	122	122,94,137,77,93
Dichlorovos	16.48	109	109,185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	120,135,134,91,77
N-Nitrosodibutylamine	16.73	84	84,57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	110,81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	110,81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	162,162,104,77,103,135
Hexamethyl phosphoramide	17.33	135	135,44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	92,127,129,65,39
Diphenylamine	17.54[a]	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	216,214,179,108,143,218
1-Naphthylamine	18.20	143	143,115,89,63
1-Acetyl-2-thiourea	18.22	118	43,118,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	174,145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	196,198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	84,133,161,162
Pentachlorophenol	19.25	266	264,268

TABLE 1.
(Continued)

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	199,152,141,169,151
Phosphamidon	26.85	127	127,264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	231,185,41,193,266
Methyl parathion	27.03	109	109,125,263,79,93
Carbaryl	27.17	144	144,115,116,201
Dimethylaminoazobenzene	27.50	225	225,120,77,105,148,42
Propylthiouracil	27.68	170	170,142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d_{12} (I.S.)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	173,125,127,93,158
Kepone	28.18	272	272,274,237,178,143,270
Fenthion	28.37	278	278,125,109,169,153
Parathion	28.40	109	109,97,291,139,155
Anilazine	28.47	239	239,241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	212,106,196,180
Carbophenothion	28.58	157	157,97,121,342,159,199
5-Nitroacenaphthene	28.73	199	199,152,169,141,115
Methapyrilene	28.77	97	97,50,191,71
Isodrin	28.95	193	193,66,195,263,265,147
Captan	29.47	79	79,149,77,119,117
Chlorfenvinphos	29.53	267	267,269,323,325,295
Crotoxyphos	29.73	127	127,105,193,166
Phosmet	30.03	160	160,77,93,317,76
EPN	30.11	157	157,169,185,141,323
Tetrachlorvinphos	30.27	329	109,329,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	223,167,195
Barban	30.83	222	222,51,87,224,257,153
Aramite	30.92	185	185,191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	283,285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	251,139,253,111,141
Fensulfothion	31.87	293	293,97,308,125,292
Ethion	32.08	231	231,97,153,125,121
Diethylstilbestrol	32.15	268	268,145,107,239,121,159
Famphur	32.67	218	218,125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	368,367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (I.S.)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	256,241,239,120
5,5-Diphenylhydantoin	33.40	180	180,104,252,223,209
Captafol	33.47	79	79,77,80,107

TABLE 1. (Continued)

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
Dinocap	33.47	69	69,41,39
Methoxychlor	33.55	227	227,228,152,114,274,212
2-Acetylaminofluorene	33.58	181	181,180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	231,266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	244,201,229
3-Methylcholanthrene	35.07	268	268,252,253,126,134,113
Phosalone	35.23	182	182,184,367,121,379
Azinphos-methyl	35.25	160	160,132,93,104,105
Leptophos	35.28	171	171,377,375,77,155,379
Mirex	35.43	272	272,237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,201,119,217,219,199
Dibenz(a,j)acridine	36.40	279	279,280,277,250
Mestranol	36.48	277	277,310,174,147,242
Coumaphos	37.08	362	362,226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	302,151,150,300
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	162,135,105,77
Hexachlorophene	47.98	196	196,198,209,211,406,408
Aldrin	--	66	263,220
Aroclor-1016	--	222	260,292
Aroclor-1221	--	190	224,260
Aroclor-1232	--	190	224,260
Aroclor-1242	--	222	256,292
Aroclor-1248	--	292	362,326
Aroclor-1254	--	292	362,326
Aroclor-1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr.)	--	172	171
2-Fluorophenol (surr.)	--	112	64

TABLE 1.
(Continued)

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
5-Nitro-o-toluidine	19.27	152	77,152,79,106,94
Thionazine	19.35	107	96,107,97,143,79,68
4-Nitroaniline	19.37	138	138,65,108,92,80,39
Phenanthrene-d_{10}(i.s.)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	168,75,50,76,92,122
Mevinphos	19.90	127	127,192,109,67,164
Naled	20.03	109	109,145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	168,76,50,75,92,122
Diallate (cis or trans)	20.57	86	86,234,43,70
1,2-Dinitrobenzene	20.58	168	168,50,63,74
Diallate (trans or cis)	20.78	86	86,234,43,70
Pentachlorobenzene	21.35	250	250,252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	168,79,52,138,153,77
Pentachloronitrobenzene	21.72	237	237,142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	174,101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	232,131,230,166,234,168
Demeton-O	22.72	88	88,89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	75,74,213,120,91,63
Dicrotophos	23.82	127	127,67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	306,43,264,41,290
Bromoxynil	23.90	277	277,279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	127,192,67,97,109
Phorate	24.10	75	75,121,97,93,260
Sulfallate	24.23	188	188,88,72,60,44
Demeton-S	24.30	88	88,60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	87,93,125,143,229
Phenobarbital	24.70	204	204,117,232,146,161
Carbofuran	24.90	164	164,149,131,122
Octamethyl pyrophosphoramide	24.95	135	135,44,199,286,153,243
4-Aminobiphenyl	25.08	169	169,168,170,115
Terbufos	25.35	231	231,57,97,153,103
a,a-Dimethylphenylamine	25.43	58	58,91,65,134,42
Pronamide	25.48	173	173,175,145,109,147
Aminoazobenzene	25.72	197	92,197,120,65,77
Dichlone	25.77	191	191,163,226,228,135,193
Dinoseb	25.83	211	211,163,147,117,240
Disulfoton	25.83	88	88,97,89,142,186
Fluchloralin	25.88	306	306,63,326,328,264,65
Mexacarbate	26.02	165	165,150,134,164,222
4,4'-Oxydianiline	26.08	200	200,108,171,80,65

TABLE 1.
(Continued)

Compound	Retention Time (min.)	Primary Ion	Secondary Ion(s)
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d_5 (surr.)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d_6 (surr.)	--	99	42,71
Terphenyl-d_{14} (surr.)	--	244	122,212
2,4,6-Tribromophenol (surr.)	--	330	332,141
Toxaphene	--	159	231,233

I.S. = internal standard.
surr. = surrogate.
[a]Estimated retention times.
[b]Substitute for the non-specific mixture, tricresyl phosphate.

TABLE 2.
ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS[a]

Semivolatiles	Estimated Quantitation Limits[b]	
	Ground water µg/L	Low Soil/Sediment[c] µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND

TABLE 2.
(Continued)

Semivolatiles	Estimated Quantitation Limits[b] Ground water µg/L	Low Soil/Sediment[1] µg/kg
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660

TABLE 2.
(Continued)

Semivolatiles	Estimated Quantitation Limits[b] Ground water µg/L	Low Soil/Sediment[1] µg/kg
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND

TABLE 2.
(Continued)

Semivolatiles	Estimated Quantitation Limits[b] Ground water µg/L	Low Soil/Sediment[1] µg/kg
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodibutylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramide	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660

TABLE 2.
(Continued)

Semivolatiles	Estimated Quantitation Limits[b] Ground water µg/L	Low Soil/Sediment[1] µg/kg
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
Toluene diisocyanate	100	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
0,0,0-Triethylphosphorothioate	NT	ND

a EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. This is based on a 30 g sample and gel permeation chromatography cleanup.
b Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable.
ND = Not determined.
NA = Not applicable.
NT = Not tested.

Other Matrices	Factor[1]
High-concentration soil and sludges by ultrasonic extractor	7.5
Non-water miscible waste	75

[1]EQL = [EQL for Low Soil/Sediment (Table 2)] X [Factor].

TABLE 3.
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a]See Reference 4.

TABLE 4.
CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5.
SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr.)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr.)
4-Methylphenol	Nitrobenzene	Hexachlorocyclo-pentadiene
N-Nitrosodimethylamine	Nitrobenzene-d_8 (surr.)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodibutylamine	2-Nitroaniline
Phenol-d_6 (surr.)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tetra-chlorophenol
		2,4,6-Tribromo-phenol (surr.)
		2,4,6-Trichloro-phenol
		2,4,5-Trichloro-phenol

(surr.) = surrogate

TABLE 5.
(Continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i) perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h) anthracene
1,2-Diphenylhydrazine	p-Dimethylaminoazobenzene	7,12-Dimethylbenz-(a)anthracene
Fluoranthene	Pyrene	Di-n-octyl phthalate
Hexachlorobenzene	Terphenyl-d_{14} (surr.)	Indeno(1,2,3-cd) pyrene
N-Nitrosodiphenylamine		3-Methylcholanthrene
Pentachlorophenol		
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr.) = surrogate

TABLE 6.
QC ACCEPTANCE CRITERIA[a]

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116

TABLE 6.
(Continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p_s (%)
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L.

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

a Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

TABLE 7.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Compound	Accuracy, as recovery, x' (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S' (μg/L)
Acenaphthene	0.96C+0.19	0.15$\bar{x}$-0.12	0.21$\bar{x}$-0.67
Acenaphthylene	0.89C+0.74	0.24$\bar{x}$-1.06	0.26$\bar{x}$-0.54
Aldrin	0.78C+1.66	0.27$\bar{x}$-1.28	0.43$\bar{x}$+1.13
Anthracene	0.80C+0.68	0.21$\bar{x}$-0.32	0.27$\bar{x}$-0.64
Benz(a)anthracene	0.88C-0.60	0.15$\bar{x}$+0.93	0.26$\bar{x}$-0.21
Chloroethane	0.99C-1.53	0.14$\bar{x}$-0.13	0.17$\bar{x}$-0.28
Benzo(b)fluoranthene	0.93C-1.80	0.22$\bar{x}$+0.43	0.29$\bar{x}$+0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19$\bar{x}$+1.03	0.35$\bar{x}$+0.40
Benzo(a)pyrene	0.90C-0.13	0.22$\bar{x}$+0.48	0.32$\bar{x}$+1.35
Benzo(ghi)perylene	0.98C-0.86	0.29$\bar{x}$+2.40	0.51$\bar{x}$-0.44
Benzyl butyl phthalate	0.66C-1.68	0.18$\bar{x}$+0.94	0.53$\bar{x}$+0.92
β-BHC	0.87C-0.94	0.20$\bar{x}$-0.58	0.30$\bar{x}$+1.94
δ-BHC	0.29C-1.09	0.34$\bar{x}$+0.86	0.93$\bar{x}$-0.17
Bis(2-chloroethyl) ether	0.86C-1.54	0.35$\bar{x}$-0.99	0.35$\bar{x}$+0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16$\bar{x}$+1.34	0.26$\bar{x}$+2.01
Bis(2-chloroisopropyl) ether	1.03C-2.31	0.24$\bar{x}$+0.28	0.25$\bar{x}$+1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26$\bar{x}$+0.73	0.36$\bar{x}$+0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13$\bar{x}$+0.66	0.16$\bar{x}$+0.66
2-Chloronaphthalene	0.89C+0.01	0.07$\bar{x}$+0.52	0.13$\bar{x}$+0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20$\bar{x}$-0.94	0.30$\bar{x}$-0.46
Chrysene	0.93C-1.00	0.28$\bar{x}$+0.13	0.33$\bar{x}$-0.09
4,4'-DDD	0.56C-0.40	0.29$\bar{x}$-0.32	0.66$\bar{x}$-0.96
4,4'-DDE	0.70C-0.54	0.26$\bar{x}$-1.17	0.39$\bar{x}$-1.04
4,4'-DDT	0.79C-3.28	0.42$\bar{x}$+0.19	0.65$\bar{x}$-0.58
Dibenzo(a,h)anthracene	0.88C+4.72	0.30$\bar{x}$+8.51	0.59$\bar{x}$+0.25
Di-n-butyl phthalate	0.59C+0.71	0.13$\bar{x}$+1.16	0.39$\bar{x}$+0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20$\bar{x}$+0.47	0.24$\bar{x}$+0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25$\bar{x}$+0.68	0.41$\bar{x}$+0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24$\bar{x}$+0.23	0.29$\bar{x}$+0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28$\bar{x}$+7.33	0.47$\bar{x}$+3.45
Dieldrin	0.82C+0.16	0.20$\bar{x}$-0.16	0.26$\bar{x}$-0.07
Diethyl phthalate	0.43C+1.00	0.28$\bar{x}$+1.44	0.52$\bar{x}$+0.22
Dimethyl phthalate	0.20C+1.03	0.54$\bar{x}$+0.19	1.05$\bar{x}$-0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12$\bar{x}$+1.06	0.21$\bar{x}$+1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14$\bar{x}$+1.26	0.19$\bar{x}$+0.35
Di-n-octyl phthalate	0.76C-0.79	0.21$\bar{x}$+1.19	0.37$\bar{x}$+1.19
Endosulfan sulfate	0.39C+0.41	0.12$\bar{x}$+2.47	0.63$\bar{x}$-1.03
Endrin aldehyde	0.76C-3.86	0.18$\bar{x}$+3.91	0.73$\bar{x}$-0.62
Fluoranthene	0.81C+1.10	0.22$\bar{x}$-0.73	0.28$\bar{x}$-0.60
Fluorene	0.90C-0.00	0.12$\bar{x}$+0.26	0.13$\bar{x}$+0.61
Heptachlor	0.87C-2.97	0.24$\bar{x}$-0.56	0.50$\bar{x}$-0.23
Heptachlor epoxide	0.92C-1.87	0.33x-0.46	0.28x+0.64

TABLE 7.
(Continued)

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Hexachlorobenzene	0.74C+0.66	0.18$\bar{x}$-0.10	0.43$\bar{x}$-0.52
Hexachlorobutadiene	0.71C-1.01	0.19$\bar{x}$+0.92	0.26$\bar{x}$+0.49
Hexachloroethane	0.73C-0.83	0.17$\bar{x}$+0.67	0.17$\bar{x}$+0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29$\bar{x}$+1.46	0.50$\bar{x}$-0.44
Isophorone	1.12C+1.41	0.27$\bar{x}$+0.77	0.33$\bar{x}$+0.26
Naphthalene	0.76C+1.58	0.21$\bar{x}$-0.41	0.30$\bar{x}$-0.68
Nitrobenzene	1.09C-3.05	0.19$\bar{x}$+0.92	0.27$\bar{x}$+0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27$\bar{x}$+0.68	0.44$\bar{x}$+0.47
PCB-1260	0.81C-10.86	0.35$\bar{x}$+3.61	0.43$\bar{x}$+1.82
Phenanthrene	0.87C+0.06	0.12$\bar{x}$+0.57	0.15$\bar{x}$+0.25
Pyrene	0.84C-0.16	0.16$\bar{x}$+0.06	0.15$\bar{x}$+0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15$\bar{x}$+0.85	0.21$\bar{x}$+0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23$\bar{x}$+0.75	0.29$\bar{x}$+1.31
2-Chlorophenol	0.78C+0.29	0.18$\bar{x}$+1.46	0.28$\bar{x}$+0.97
2,4-Dichlorophenol	0.87C-0.13	0.15$\bar{x}$+1.25	0.21$\bar{x}$+1.28
2,4-Dimethylphenol	0.71C+4.41	0.16$\bar{x}$+1.21	0.22$\bar{x}$+1.31
2,4-Dinitrophenol	0.81C-18.04	0.38$\bar{x}$+2.36	0.42$\bar{x}$+26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10$\bar{x}$+42.29	0.26$\bar{x}$+23.10
2-Nitrophenol	0.07C-1.15	0.16$\bar{x}$+1.94	0.27$\bar{x}$+2.60
4-Nitrophenol	0.61C-1.22	0.38$\bar{x}$+2.57	0.44$\bar{x}$+3.24
Pentachlorophenol	0.93C+1.99	0.24$\bar{x}$+3.03	0.30$\bar{x}$+4.33
Phenol	0.43C+1.26	0.26$\bar{x}$+0.73	0.35$\bar{x}$+0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16$\bar{x}$+2.22	0.22$\bar{x}$+1.81

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{x}$, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{x}$, in µg/L.

C = True value for the concentration, in µg/L.

$\bar{x}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

TABLE 8.
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
Nitrobenzene-d_5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-d_{14}	33-141	18-137
Phenol-d_6	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122

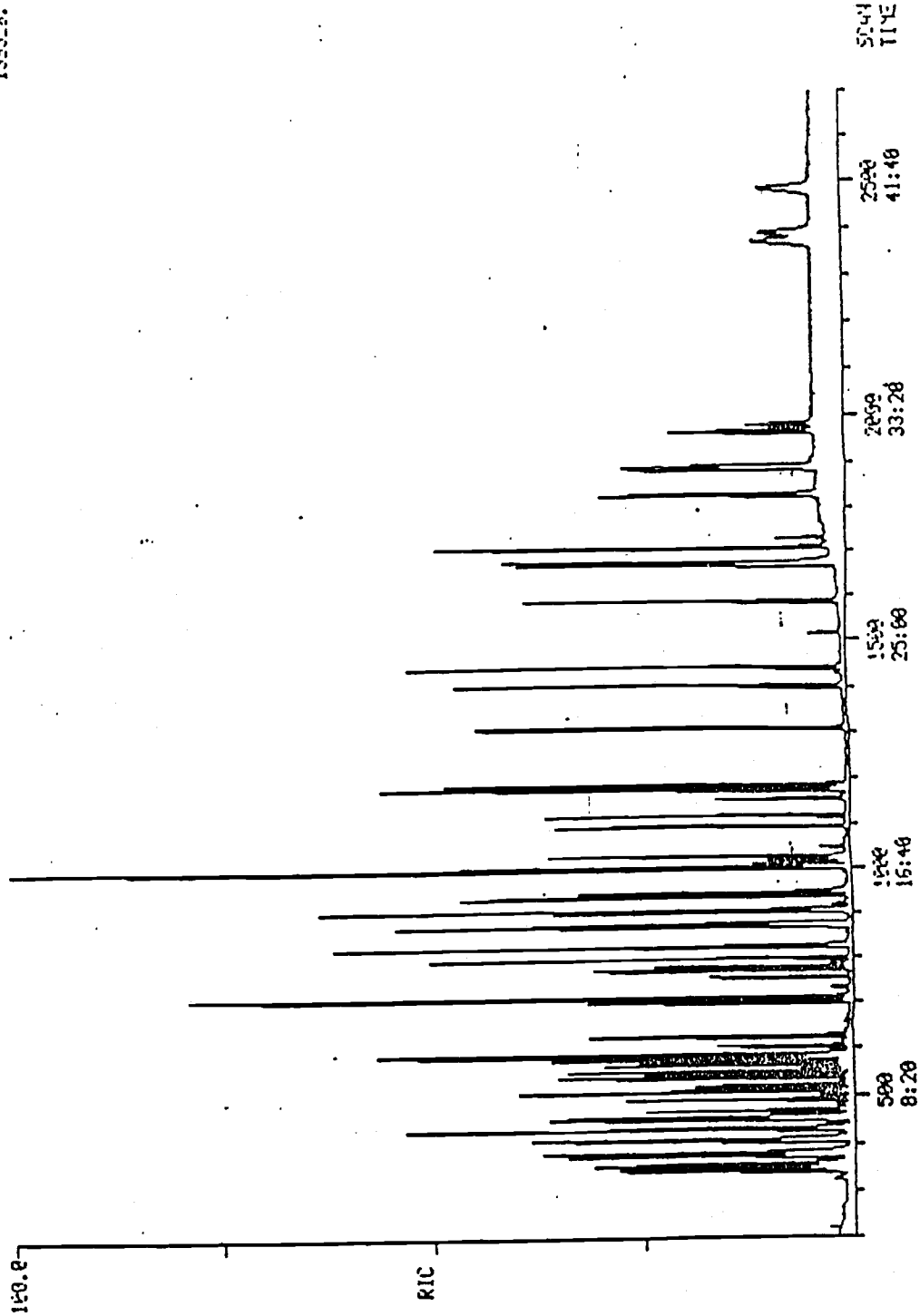
TABLE 9.
CHEMRON GENERATED
% RECOVERY CONTROL LIMITS

COMPOUND	SOIL MATRIX	WATER MATRIX
1,2,4-TCB	15-104	25-109
1,4-Dichlorobenzene	d-96	12-112
2-Chlorophenol	11-98	6-111
2,4-Dinitrotoluene	26-100	33-109
Chloromethylphenol	8-109	16-110
4-Nitrophenol	d-158	d-119
Acenaphthene	11-115	25-111
N-Nitroso-di-N-propylamine	17-108	18-123
Pentachlorophenol	d-122	18-113
Phenol	3-109	2-87
Pyrene	d-143	24-128
2-Fluorobiphenyl	23-120	43-116
2-Fluorophenol	25-121	21-100
2,4,6-Tribromophenol	19-97	10-123
Nitrobenzene, d5	36-92	35-114
Phenol, d5	24-113	10-94
Terphenyl, d14	18-137	33-135

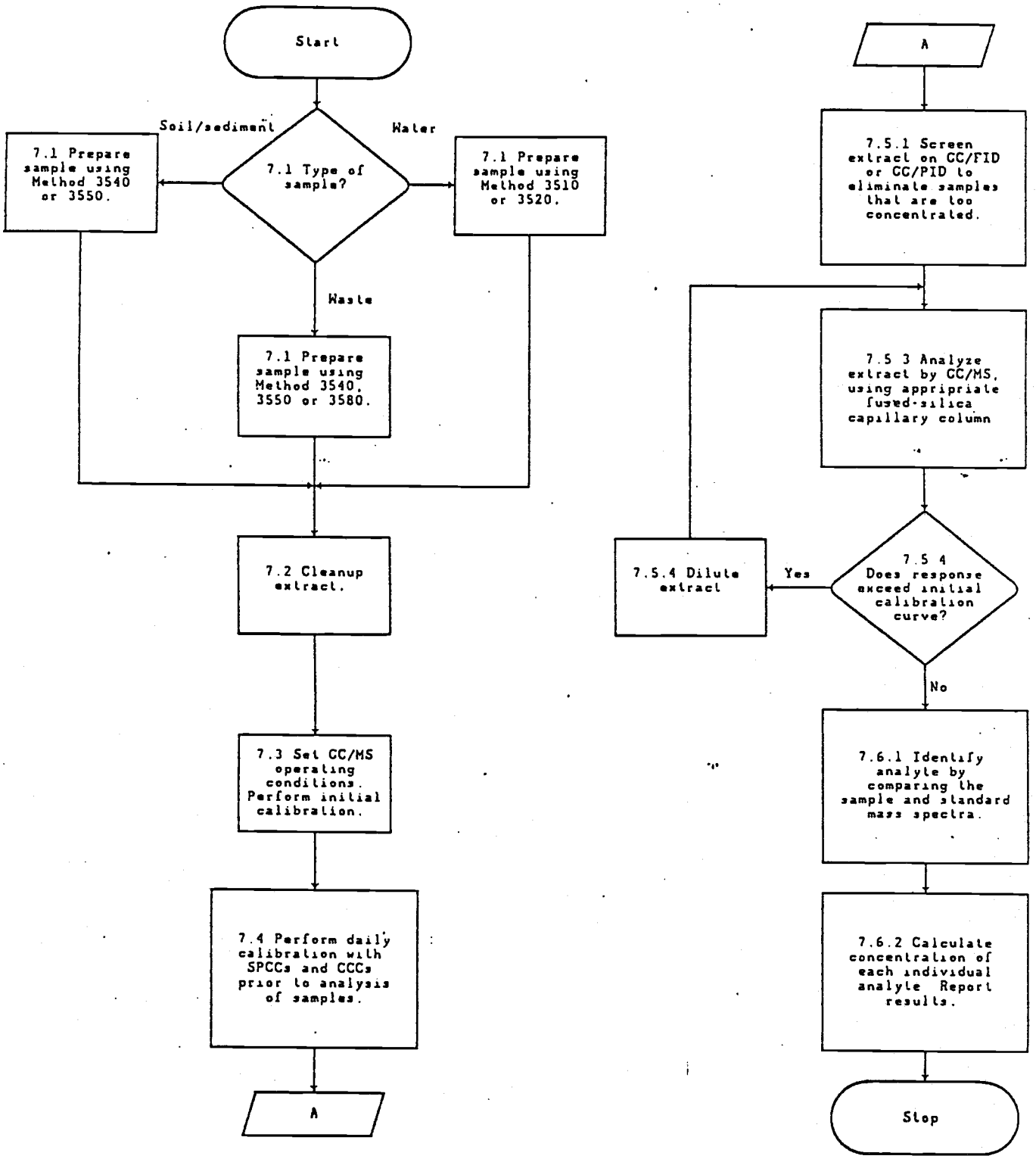
TABLE 10.
CHEMRON ACCURACY ASSESSMENT INTERVALS

COMPOUND	SOIL MATRIX	WATER MATRIX
1,2,4-TCB	30-89	40-95
1,4-Dichlorobenzene	10-79	29-96
2-Chlorophenol	25-83	24-94
2,4-Dinitrotoluene	38-88	45-96
Chloromethylphenol	25-93	31-94
4-Nitrophenol	d-126	2-96
Acenaphthene	28-98	39-97
N-Nitroso-di-N-propylamine	32-93	35-106
Pentachlorophenol	d-97	33-97
Phenol	20-91	16-73
Pyrene	23-119	41-111
2-Fluorobiphenyl	26-143	35-99
2-Fluorophenol	22-105	16-87
2,4,6-Tribromophenol	11-97	8-107
Nitrobenzene, d5	45-83	19-100
Phenol, d5	36-106	9-91
Terphenyl, d14	39-127	38-136

FIGURE 1.
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE



CHEMRON INC. - STANDARD OPERATING PROCEDURE 9060

TOTAL ORGANIC CARBON

1.0 SCOPE AND APPLICATION

1.1 Standard Operating Procedure (SOP) 9060 is used to determine the concentration of organic carbon in ground water, surface and saline waters, and domestic and industrial wastes. Some restrictions are noted in Sections 2.0 and 3.0.

1.2 SOP 9060 is most applicable to measurement of organic carbon above 1 mg/L.

2.0 SUMMARY OF METHOD

2.1 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO_2) by either catalytic combustion or wet chemical oxidation. The CO_2 formed is then either measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.2 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of preliminary sample treatment as well as the instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by SOP 9060 are:

1. Soluble, nonvolatile organic carbon: e.g., natural sugars.

2. Soluble, volatile organic carbon: e.g., mercaptans, alkanes, low molecular weight alcohols.

3. Insoluble, partially volatile carbon: e.g., low molecular weight oils.

4. Insoluble, particulate carbonaceous materials: e.g., cellulose fibers.

5. Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g.,oily matter adsorbed on silt particles.

2.3 Carbonate and bicarbonate are inorganic forms of carbon and must be separated from the total organic carbon value. Depending on the instrument manufactuer's instructions, this separation can be accomplished by either a simple mathematical subtraction, or by removing the carbonate and bicarbonate by converting them to CO_2 with degassing prior to analysis.

3.0 INTERFERENCES

3.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

3.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a microliter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle which may be included in the sample.

3.3 Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

4.2 Apparatus for total and dissolved organic carbon:

4.2.1 Several companies manufacture analyzers for measuring carbonaceous material in liquid samples. The most appropriate system should be selected based on consideration of the types of samples to be analyzed, the expected concentration range, and the forms of carbon to be measured.

4.2.2 No specific analyzer is recommended as superior. If the technique of chemical oxidation is used, the laboratory must be certain that the instrument is capable of achieving good carbon recoveries in samples containing particulates.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities, and should be boiled and cooled to remove CO_2.

5.2 Potassium hydrogen phthalate, stock solution, 1,000 mg/L carbon: Dissolve 0.2128 g of potassium hydrogen phthalate (primary standard grade) in Type II water and dilute to 100.0 mL.

NOTE 1: Sodium oxalate and acetic acid are not recommended as stock solutions.

5.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with Type II water.

5.4 Carbonate-bicarbonate, stock solution, 1000 mg/L carbon: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100-mL volumetric flask. Dissolve with Type II water.

5.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to Step 5.3.

NOTE 2: This standard is not required by some instruments.

5.6 Blank solution: Use the same Type II water as was used to prepare the standard solutions.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

NOTE 3: A brief study performed in the EPA Laboratory indicated that Type II water stored in new, 1-qt cubitainers did not show any increase in organic carbon after 2 weeks' exposure.

6.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between sample collection and the start of analysis should be minimized. Also, samples should be kept cool (4^0C) and protected from sunlight and atmospheric oxygen.

6.3 In instances where analysis cannot be performed within 2 hours from time of sampling, the sample is acidified (pH $\leq$ 2) with HCl or H_2SO_4.

7.0 PROCEDURE

7.1 Homogenize the sample in a blender.

NOTE 4: To avoid erroneously high results, inorganic carbon must be accounted for. The preferred method is to measure total carbon and inorganic carbon and to obtain the organic carbon by subtraction. If this is not possible, follow Steps 7.2 and 7.3 prior to analysis; however, volatile organic carbon may be lost.

7.2 Lower the pH of the sample to 2.

7.3 Purge the sample with nitrogen for 10 min.

7.4 Follow instrument manufacturer's instructions for calibration procedure, and calculations.

7.5 For calibration of the instrument, a series of standards should be used that encompasses the expected concentration range of the samples.

7.6 Quadruplicate analysis is required. Report both the average and the range.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Verify calibration with an independently prepared check standard every 15 samples.

8.4 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

9.0 METHOD PERFORMANCE

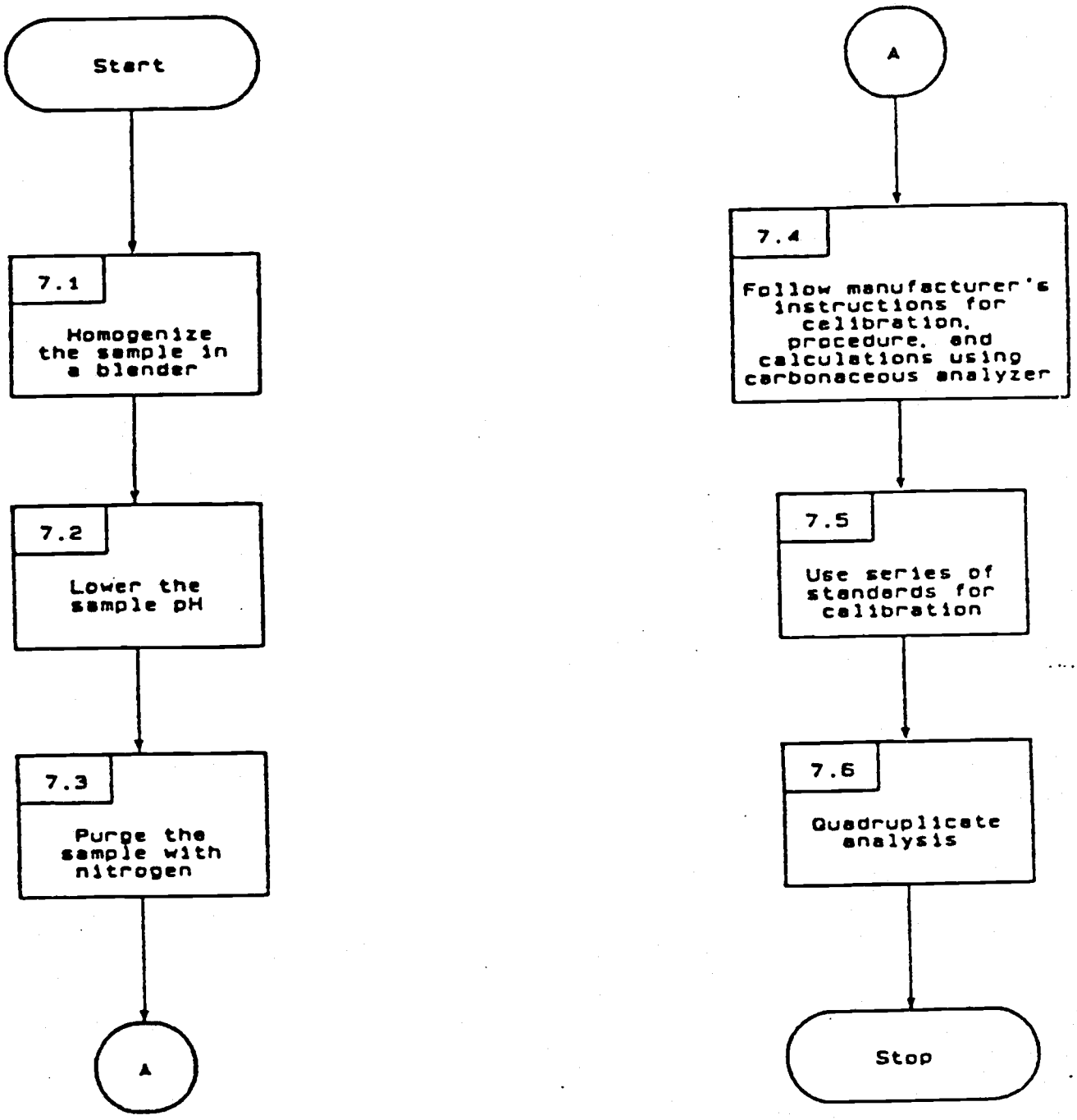
9.1 Precision and accuracy data are available in EPA Method 415.1 of Methods for Chemical Analysis of Waters and Wastes.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water" Standard D 2574-79, p. 469 (1976)
2. Standard Methods for the Examination of Water and Wastewater, 14th ed., p. 532, Method 505 (1975).
3. Test Methods for Evaluating Solid Waste Physical/ Chemical Methods, SW-846, 3rd Ed.,ECR, Inc. Method 9060 (1986).
4. "Methods for Chemical Analysis of Water and Wastes," EPA Method 415.1,EPA-6001/4-79-020.

Date - November 23, 1994

Approved: _____ Date: 12/06/94


Start
7.1
Homogenize the sample in a blender
7.2
Lower the sample pH
7.3
Purge the sample with nitrogen
A
A
7.4
Follow manufacturer's instructions for calibration, procedure, and calculations using carbonaceous analyzer
7.5
Use series of standards for calibration
7.6
Quadruplicate analysis
Stop

CHEMRON
INCORPORATED

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QUALITY ASSURANCE PLAN

MARCH, 1994

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1.0 INTRODUCTION

The management of Chemron Incorporated is committed to providing analytical data of the highest quality. To meet this objective Chemron has implemented a Quality Assurance Plan (QAP) that establishes procedures for demonstrating that analytical systems are in control and provides a means for measuring the quality of all analytical data. This QAP documents the quality assurance (QA) and quality control (QC) policies and practices conducted by Chemron for all laboratory work.

This document provides a detailed description of the policies, organization, and QA and QC protocols that have been implemented to achieve the data quality needs of each project. The following topics are summarized in the QAP:

- Laboratory Organization
- Quality Assurance Objectives
- Corrective Action
- Sample Preservation & Holding Times
- Sample Custody
- Analytical Procedures
- Internal Quality Control Checks
- Data Assessment Procedures

2.0 LABORATORY ORGANIZATION

Chemron's organizational structure is presented in Figure 2-1. The responsibilities to ensure that quality goals are achieved for all programs are described in this section.

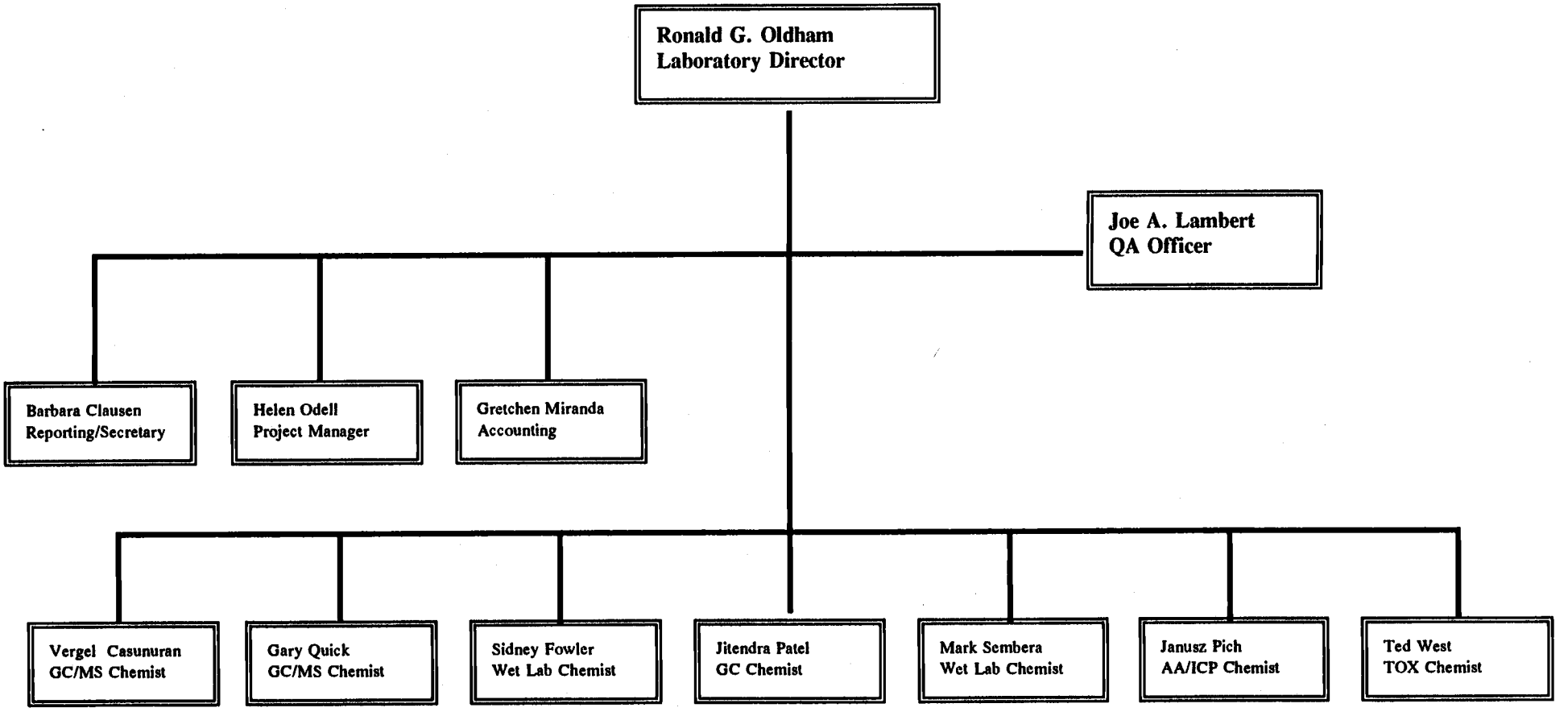
Within Chemron, the Quality Assurance Director, Mr. Joe Lambert, has the responsibility for implementing and monitoring Chemron's QA program. Mr. Lambert has sufficient authority and freedom to:

- Identify problems affecting quality
- Initiate, recommend and provide solutions to analytical problems.
- Verify the implementation of corrective actions.
- Ensure that further work is stopped until the proper resolution of a nonconformance, deficiency or unsatisfactory condition has occurred.

The ultimate responsibility for the generation of reliable analytical data resides with the Laboratory Director, Mr. Ronald Oldham. Mr. Oldham has the authority to implement policies and procedures to ensure that data of the highest quality are produced. It is also Mr. Oldham's responsibility to see that all tasks performed in the laboratory are conducted according to the requirements of Chemron's QAP or any additional program specific requirements.

Figure 2-1

CHEMRON ORGANIZATIONAL CHART



3.0 QUALITY ASSURANCE OBJECTIVES

The overall objective for laboratory analyses is to provide reliable data of known and documentable quality, ensuring that the sample holding times were not violated. Qualitative and quantitative statements, referred to as data quality objectives (DQOs), are used to describe the level of quality required for a specific task. A means to measure the attainment of stated DQOs is through the use of five characteristics: precision, accuracy, representativeness, comparability, and completeness. Tables 3-1, 3-2, and 3-3 list the QA objectives for selected organic and inorganic analyses.

3.1 Accuracy

Accuracy is the degree of agreement of the analytical measurement with the true or expected concentration. Analytical accuracy is expressed as the percentage recovery (%R) of analyte that has been used to fortify an investigative sample or a standard matrix (e.g., blank soil, analyte-free water, etc.) at a known concentration prior to analysis and is expressed by the following formula:

$$\text{Accuracy} = \%\ \text{Recovery}\ (\%R) = \frac{A_T - A_o}{A_F} \times 100\%$$

Where:

AT = total amount found in fortified sample
AO = amount found in unfortified sample
AF = amount added to sample

Accuracy control limits are established and controlled by the Laboratory Control Samples (LCS). The %R of the spiked analysis is recorded and evaluated against statistically generated control limits. The upper and lower control limits are based on $\pm$ 3 times the standard deviation of the mean of the accumulated LCS results. Corrective action for out-of-control situations is discussed in sections 3.2 and 4.0 and summarized in Table 9-1.

3.2 Precision

Precision is the level of agreement among repeated independent measurements of the same characteristic, usually under a prescribed set of conditions. The most commonly used estimates of precision are the relative percentage difference (RPD) used when only two measurements are available, and the percent relative standard deviation (% RSD) used when three or more measurements are available.

$$RPD = \frac{(C_1 - C_2)}{(C_1 + C_2) / 2} \times 100\%$$

Table 3-1
Quality Assurance Objectives
for Inorganic Analyses Accuracy and Precision

Sample Type	Recovery (%)	% RPD
Laboratory Control Sample (LCS)	80-120*	≤20
Fortified Field Sample	75-125	≤20

Ag = 50 - 120(s) & 60 - 120(w)
Sb = 60 - 120(soil and water)

Control limits are updated on a routine basis.

Where:

RPD = relative percent difference
C_1 = concentration of analyte in sample
C_2 = concentration of analyte in replicate

The %RSD is calculated by expressing as a percentage the standard deviation of the analytical results of the replicate determinations relative to the average of those results for a given analyte. Acceptance criteria for %RSD are interchangeable with those established for %RPD. This method of precision measurement can be expressed by the formula:

$$\%RSD = \frac{[(\Sigma C^2)\, n - (\Sigma C^2)]/n(n-1))^{1/2}}{(C_1 + C_2 + \ldots C_n)/n} \times 100\%$$

Where:

%RSD	=	percent relative standard deviation
C	=	concentration of analyte in the sample
$(C_1 + C_2 + \ldots C_n)$	=	the sum of the concentration of replicate
n	=	the number of replicate analyses
Σ	=	the summation of

Precision control limits are established and controlled by the duplicate analysis of LCS. The LCS may be purchased commercially or prepared at the laboratory and may also be identified as blank spikes (BS). For multi-analyte methods, including preparation methods such as metals digestion, the LCS may contain only a representative number of target analytes rather than the full list. For organic analyses, the LCS pair may be surrogate compounds in the blank and the blank spike and/or a select number of target analytes in duplicate fortified blanks (e.g., blank/blank spike duplicate: BS/BSD). The RPD of the duplicate analysis is recorded and evaluated against statistically generated control limits. The maximum allowable RPD control limit is based on the average of the accumulated RPDs plus three times the standard deviation of the accumulated RPDs of the duplicate LCS results.

When the LCS %R or RPD exceeds the established acceptance limits, appropriate corrective action is taken. After the problem has been identified and corrected and control has been reestablished, sample analysis may continue. All data associated with the out-of- control situation are evaluated with respect to project DQOs for usability and sample availability for reanalysis. For rejected results, the samples are reprepared and/or reanalyzed after control has been reestablished. If data are used without reanalysis, the case narrative will address the deviation.

3.3 Completeness

Completeness is a measure of the relative number of analytical data points that meet all the

acceptance criteria for accuracy, precision, and any other criteria required by the specific analytical methods used. Project specific completeness goals account for all aspects of sample handling, from collection through data reporting. Completeness goals for collections of analytical data (e.g., groups of samples, analytical batches, analytes within a method, etc.) is typically 90%.

3.4 Representativeness and Comparability

Laboratory procedures will ensure that all data are representative of the matrix and conditions of the sample being measured. The data will be calculated and reported in units consistent with the requirements of the project.

4.0 CORRECTIVE ACTION

Immediate corrective action for the repair of nonconforming laboratory equipment and systems is generally initiated as the result of QC procedures. Quick feedback that a problem exists (e.g., calibration does not meet standards or QC check samples exceed allowable criteria) allow immediate action to repair the system.

Long-term corrective action is generally initiated because of QA issues. These issues are most often identified during audits. Long-term corrective action involves a deeper investigation into the cause of the nonconformance, and it may take much longer to identify and resolve nonconformances. Staff training, method revision, replacement of equipment, or Laboratory Information Management System (LIMS) reprogramming may be part of a long-term corrective actions.

All corrective actions, whether immediate or long term, will include the following steps to ensure a closed-loop corrective action system:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminate the problem.

The analyst will have the initial responsibility to monitor the quality of an analytical system. The analyst will verify that all QC procedures are followed and the results of the analysis of QC samples are within acceptance criteria. The analyst is required to assess the correctness of all the following items as appropriate:

- Sample preparation procedure.
- Initial calibration.
- Calibration verification.
- Method blank result.
- Laboratory control standard.
- Duplicate analysis.
- Fortified sample result.

If the assessment reveals that any of the QC acceptance criteria are not met, the analyst will immediately assess the analytical system to correct the problem. When an acceptable resolution cannot be met and/or date quality is negatively impacted, the analyst will notify the appropriate supervisor and complete a Corrective Action Form (see Figure 4-1). If possible, potential causes and corrective action will be identified. The Managers will be

Figure 4-1

CORRECTIVE ACTION FORM

Date:_____

Analyst:_____

Analysis:_____

Brief description of quality control failure:_____

_____

_____

Sample #(s)	% Recovery	% Precision	Comments
1st _____	_____	_____	_____
2nd _____	_____	_____	_____
3rd _____	_____	_____	_____

Corrective Action Planned / Remarks:_____

_____

_____

_____

_____

Approved by:_____ Date:_____

responsible for correcting out-of-control situations and for placing the highest priority on this endeavor.

The nature of the corrective action will depend on the nature of the problem. For example, if a continuing calibration verification is determined to be out of control, the corrective action may require recalibration of the analytical system and reanalysis of all samples since the last acceptable continuing calibration standard. When the appropriate corrective action measures have been defined and the analytical system is determined to be in control, the analyst will document the problem and the corrective action. Copies of the form summarizing these actions will be provided to the QA Manager. A summary of common corrective actions can be found in Table 9-1.

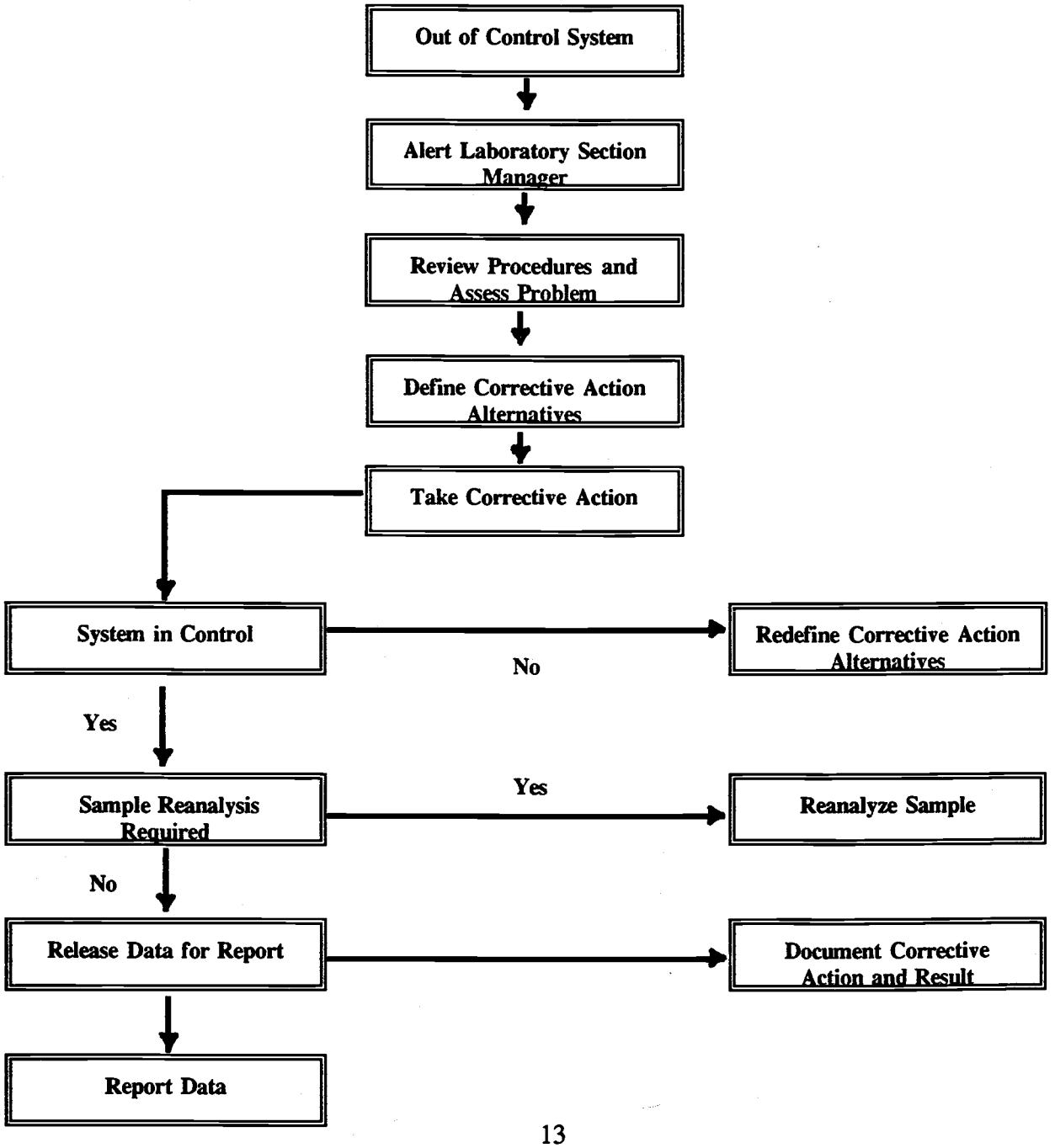
Data generated with an out-of-control system will be evaluated for usability. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired, the Laboratory Project Manager will be notified and appropriate corrective action (e.g., reanalysis) taken.

The critical path for assessing the correctness and acceptability of analytical data is shown in Figure 4-2. The QA Manager has the authority to stop the analysis and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without the appropriate documentation leading to the QA Manager's approval to restart the method.

A summary of the results from external on-site audits or performance evaluations studies will be distributed to appropriate laboratory personnel. The person responsible for the resolution of the deficiency will issue a memo addressing the findings and resultant steps to correct the problem. The QA Manager will then forward the corrective action information to the respective outside client or agency.

Figure 4-2

CRITICAL PATH FOR CORRECTIVE ACTION



5.0 LABORATORY DOCUMENTATION

Data related to all sample preparation and analysis procedures and observations by laboratory analysts will be recorded in bound laboratory notebooks that are issued by the QA Manager. These notebooks may be pre-formatted bench sheets or analysis run logs, which will be bound into notebooks, or hardbound scientific notebooks, which will be formatted by the analyst.

Data will be recorded and associated with a unique Chemron sample identification number for internal tracking and reporting. Notebook pages may contain the following information, as applicable: analytical method, analyst, date, logbook number, sequential page number, associated sample numbers, reagent concentrations, instrument settings, and raw data.

Laboratory notebook pages will be signed and dated daily by the laboratory analysts. The notebook pages will be reviewed periodically by the Section Manager or designee. Copies of instrument outputs (chromatograms, strip charts, etc.) will be maintained on file.

All raw data, such as hardbound laboratory notebooks and logbooks, strip charts and instrument printouts, spreadsheets, and magnetic tapes, as well as final reports, will be retained for a minimum period of 5 years. These data and reports will be documented and stored in a manner that allows for easy retrieval. Prior to transfer to Chemron's secure archives area, interim storage of raw data records will be maintained in the laboratory. All hardbound laboratory notebooks and logbooks will be assigned a book number by the QA Section. New logbooks will be issued for each instrument or parameter when the most current book has been completely filled.

6.0 SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

As general guidance, all sample containers provided by the laboratory should be filled. This will provide adequate volumes to perform each analysis. Special consideration will be given to the type of sample container and preservative needed for each analyte when determining the sample volume required for each location. Analytes with the same container/ preservative requirement may be combined into one larger container.

All sample containers provided by Chemron will be shipped with chain-of-custody sheets, completed by the field sampling personnel and returned with the samples.

The preservatives required for all analyses will be provided with the sample containers by Chemron. The required preservation methods (e.g., pH, chilled, etc.) will be in accordance with EPA, USACE or TNRCC requirements. A summary of required sample containers, sample volumes, preservation, and holding times is presented in Table 6-1.

Sample preservation will be checked upon receipt of samples at the laboratory, and this information will be recorded on the chain-of-custody form submitted with the sample (Figure 1-11). Preservation for volatile organic compounds (VOCs) will be verified at the time of analysis.

Holding time is defined as the maximum time that may elapse before sample analysis or preparation for analysis is begun. Holding times are measured from the time of collection unless otherwise specified. Holding times will be tracked in the laboratory with the aid of the Laboratory Information Management System (LIMS). All sample collection dates and receipt dates will be recorded on the chain-of-custody. This information will be transferred into LIMS by sample log-in-personnel. Additionally, log-in personnel will enter holding time information (i.e., from collection or from receipt) into LIMS, based on the information provided on the chain-of-custody document. LIMS will calculate the appropriate holding time for each parameter. This information then will be available to the analytical personnel for tracking and scheduling purposes. Due dates shown for the analysis (or extraction) of each parameter on the LIMS backlog reports will be based on the required reporting date, or on the holding time if it falls sooner than the required reporting date for analyses.

Table 6-1. Requirements for Containers, Preservation Techniques, Sample Volumes and Holding Times

Name	Method of Analysis	Container [1]	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
INORGANIC TESTS					
Alkalinity	A403	P,G	Cool, 4°C	N/A	Analyze Immediately
Common Anions	E300	P,G	None Required	50 ml	28 Days for Br,F,Cl,SO_4
Cyanide, Total, & Amenable to Chlorination	SW9010	P,G,T	Cool, 4°C, NaOH to pH.12[2] 0.6 g ascorbic acid	500 ml or 4 ounces	14 days (water and soil)
Filterable Residue	E160.1	P,G	Cool, 4°C	100 ml	7 days
Non-Filterable Residue	E160.2	P,G	Cool, 4°C	100 ml	7 days
Hydrogen Ion (pH)	SW9040	P,G	None Required	N/A	Analyze Immediately
Nitrogen, Nitrate+Nitrite	E353.2	P,G	Cool,4°C ,H_2SO_4 to pH<2[2]	500 ml	28 days
Specific Conductance	SW9050	P,G	Cool, 4°C	N/A	Analyze Immediately
Temperature	E170.1	P,G	None Required	N/A	Analyze Immediately
Total Organic Carbons	SW9060	P,G,T	Cool,4°C,H_2SO_4 to pH<2[2]	500 ml or 4 ounces	28 days (water and soil)
METALS					
Chromium VI	SW7196	P,G,T	Cool, 4°C	500 ml or 8 ounces	24 hours (water and soil)
Mercury	SW7470/SW7471	P,G,T	HNO_3 to pH<2,[2] Cool, 4°C	500 ml or 8 ounces	28 days (water and soil)

Table 6-1. Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times (continued)

Name	Method of Analysis	Container [1]	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
METALS, Con't.					
Metals, except Chromium VI and Mercury	SW6010 and SW846 atomic absorption methods	P,G,T	HNO_3 to pH<2,[2] Cool, 4°C	500 ml or 8 ounces	180 days (water and soil
ORGANIC TESTS					
Petroleum Hydrocarbons	E418.1	G,T	Cool,4°C,H_2SO_4 to pH<2[2]	1 liter or 8 ounces	Water - 28 days Soil - 14 days until extraction 40 days after extraction
Fuel Hydrocarbons	SW8015 (modified),GC-FID[3]	G, teflon-lined Septum, T	Cool,4°C,H_2SO_4 to pH<2[2]		Volatiles - 14 days Semivolatiles - 14 days until extraction, 40 days after extraction
Aromatic Volatile Organics	SW8020	G, teflon-lined Septum, T	Cool,4°C,HI to pH <2,[2] 0.008% $Na_2S_2O_2$	2x40 ml or 4 ounces	14 days (water and soil) 7 days unpreserved by acid
Chlorinated Herbicides	SW8150	G, teflon-lined Cap, T	Cool,4°C pH 5-9	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction
Pesticides and PCB	SW8080/SW8140	G, teflon-lined Cap, T	Cool,4°C pH 5-9	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction

Table 6-1. Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times (continued)

Name	Method of Analysis	Container [1]	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
Semivolatile Organics	SW8240	G, teflon-lined Cap, T	Cool,4°C 0.008% $Na_2S_2O_2$	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction
ORGANIC TESTS, Con't.					
Volatile Organics	SW8240/SW8015/ SW8010	G, teflon-lined Septum, T	Cool,4°C 0.008% $Na_2S_2O_2$(HCL to pH<2 for volatile aromatics by SW8240)[2]	2x40 ml or 4 ounces	14 days (water and soil) 7 days unpreserved by acid
Polycyclic Aromatic Hydrocarbons (PAHs)	SW8310	G, teflon-lined Cap,T	Cool,4°C, Store in Dark, 0.008% $Na_2S_2O_2$	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction
Carbamate Pesticides	SW8318	G, teflon-lined Cap, T	Cool,4°C 0.008% $Na_2S_2O_2$	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction
Dioxins	SW8318	G, teflon-lined Cap, T	Cool,4°C 0.008% $Na_2S_2O_2$	1 liter or 8 ounces	Water - 7 days until extraction 40 days after extraction Soil - 14 days until extraction 40 days after extraction

Table 6-1. Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times (continued)

Name	Method of Analysis	Container [1]	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
RADIOLOGICAL TESTS					
Alpha, Beta, and Radium	SW9310/SW9315	P,G,T	HNO_3 to pH<2[2]	2 liters or 16 ounces	180 days
MISCELLANEOUS					
Toxicity Characteristic Leaching Procedure (TCLP)	SW1311	G, teflon-lined Cap, T	Cool, 4°C	1 liter or 8 ounces	Volatiles-14 days to TCLP extraction 14 days after extraction Semivolatiles-14 days to TCLP extraction, 7 days to prep extraction, 40 days after prep extrac tion Mercury-28 days to TCLP extraction 28 days after extraction Metals-180 days to TCLP extraction 180 days after extraction
Explosive Residues	SW8313	P,G,T	Cool, 4°C	1 liter or 8 ounces	Water and soils - 56 days to extrac tion, 40 days after extraction

[1] Polyethylene (P), Glass (G), California brass (T)
[2] No pH adjustment for soil
[3] Gas Chromatography-Flame Ionization Detection (GC-FID)

7.0 SAMPLE CUSTODY

The purpose of chain-of-custody procedures is to document the history of sample containers and samples, including sample extracts or digestates. The associated records provide traceability from the time of preparation of sample containers, through collection, shipment, analysis, and disposal of the sample. Items under custody will be:

- Maintained in the physical possession or view of the responsible party, or
- Placed and/or stored in a designated secure area to prevent tampering. This secure area will be accessible only to authorized personnel.

7.1 Laboratory Custody

A designated Sample Custodian will be responsible for samples received at the Chemron laboratories. The sample custodian will be also responsible for documenting sample receipt, storage before and after sample analysis, and the eventual proper disposal of samples.

Upon receipt, the Sample Custodian will inspect the sample container for integrity. Problems with the sample containers will be noted on the chain-of-custody form. The Sample Custodian will verify sample identify and resolve with the field sampling team any inconsistencies with the chain-of-custody.

The Sample Custodian will assign a unique sample number to each sample received. The sample number will be recorded on the chain-of-custody form and the sample bottles. Samples will be logged into a sample logbook and into the LIMS. Samples will be moved to one of the sample storage refrigerators, which are maintained at 4° ($\pm$2°)C.

The Sample Custodian will maintain the original of the chain-of-custody form in the sample log-in area, and copies will be distributed to the analytical personnel for processing the sample analyses.

The samples will be tracked from sample receipt through sample disposal by the unique sample number and by using the LIMS.

Data related to sample manipulation/preparation/analysis procedures and observations will be documented by the analyst/technician in the sample extraction log, sample digestion log, sample distillation log, analysis log, or the technician's personal logbook.

7.2 Sample Storage

Samples are maintained in storage in locked storage refrigerators prior to sample preparation and analysis.

The storage refrigerators are maintained at 4° (±2°)C. The temperature is monitored by the laboratory personnel and recorded daily in a bound log by the Sample Custodian.

All samples will be help a minimum of 30 days after the data report is submitted to the client. Samples may be held longer due to special requests or specific contract requirements. All hazardous samples will either be disposed of commercially or returned to the client.

7.3 Sample Tracking

For samples that require extraction prior to analysis, a sample extraction form will be completed during the time of extraction (Figure 7-1).

When samples are extracted for analysis by gas chromatography, GC/MS, or liquid chromatography, all pertinent data will be entered on the sample extraction form and recorded in a bound laboratory notebook. As the extraction proceeds data will be entered into the LIMS. The bound laboratory notebook will be kept in the laboratory. Extracts will be maintained in refrigerated storage by the sample preparation section until transferred to the analysts.

Samples will be digested prior to analysis for metals by atomic absorption spectroscopy or inductively coupled plasma spectroscopy. When samples are prepared for digestion, the preparation technician will record the pertinent information in a laboratory digestion notebook. This information will then be transferred to the LIMS. The original copy of the sample digestion record will be retained by the Metals Sample Preparation Section.

7.4 Building Security

The Chemron laboratory maintains controlled building access at all times. During working hours, all non-Chemron laboratory personnel are required to sign in with the receptionist and are escorted by laboratory personnel while in the building.

The laboratory is locked between the hours of 5:00 P.M. and 8:00 A.M. Monday through Friday and during nonworking hours. A Security System monitors building access. The building is accessed by laboratory employees during nonworking hours by using a key and the pass code for the building Security System.

Figure 7-1

SAMPLE EXTRACTION FORM

ANALYST:_____ **DATE:**_____

LAB ID	MATRIX (Aqueous, Solid Sludge)	Amount Used (g or mL)	Extract Volume (mL)	Spike Used (mL)	Surr. Added (mL)	Analytical Method	REMARKS (Extraction method; cleanup method; pH; % solid, other observations)

Extraction Methods:
EPA 3510 (SF); EPA3520 (Cont.); EPA 3540 (SXH);
EPA 3550 (Son.); EPA 3580 (Waste Dil.)

Cleanup Methods:
EPA 3610 (Alum); EPA 3611 (lum & WD); EPA 3620(Flor);
EPA 3630 (Silgel); EPA 3640 (GPC); EPA 3650 (A/B Part.);
EPA 3660 (Sulfur)

Additional Comments:

8.0 ANALYTICAL PROCEDURES

Analytical methods that are commonly used in Chemron's laboratories are specified in Table 8-1. Table 8-1 also summaries the typical detection limits achieved for the various analytical methods.

8.1 Method References

The most commonly used method references for the analytical procedures used in the laboratory follow. The references are applicable to the analytical test methods used on a daily basis in the laboratory.

ASTM = *Annual Book of ASTM Standards*, American Society for Testing and Materials, updated yearly.

CLP-O = EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis, Multimedia, Multiconcentration: 2/88 updated through 5/89; and EPA-CLP SOW OLM01.0 (3/90) and as updated.

E = EPA 600/4-79-020, *Methods for Chemical Analysis of Water and Wastes*, March 1989.

NIOSH = NIOSH *Manual of Analytical Methods*, 3rd Edition, February 1984, updated through Supplement 4, August 1990.

SW = EPA SW-846, *Test Methods for Evaluating Solid Waste*, 3rd Edition, November 1986.

SM = *Standard Methods for the Examination of Water and Wastewater*: 15th Edition, 1980; 16th Edition, 1985; and 17th Edition, 1989 (Note method numbers changed format with 17th Edition).

THAMA = USATHAMA PAM 11-41, *United States Army Toxic and Hazardous Materials Agency (USATHAMA) Quality Assurance Program*, Revision 0, January 1990.

8.2 Detection Limits: Terminology and Procedures

The method detection limit is determined annually and is the lowest concentration that can be seen for a given analytical method and sample matrix with a 99% confidence level. The MDL is determined according to Appendix B of 40 CFR Chapter 1, Part 136 - Guidelines Establishing Test Procedures for the Analysis of Pollutants. A standard deviation and calculated 99% confidence level are determined by analysis of a minimum of seven predetermined low standards of a given matrix that have gone through the same preparation and analysis as a sample of the same matrix. MDLs reflect a calculated value determined under

Table 8-1. Maximum Quantitation Limits

	METHOD		MAXIMUM QUANTITATION LIMITS		
Parameter	W-Water S-Soil	Analyte	Water	Soil/Sediment (mg/kg)(f)	
Alkalinity	A403(w)	Carbonate Bicarbonate Hydroxide	10 10 10	mg/L mg/L mg/L	- - -
Gross Alpha & Gross Beta Radioactivity (Total suspended & dissolved)	SW9310		4	pCi/L (a)	(b)
Radium	SW9315		1	pCi/L (a)	(b)
Residue, Filterable Residue, Nonfilterable	E160.1 (W) E160.2(w)	Total Dissolved Solids Total Suspended Solids	10 5	mg/L mg/L	- -
Common Anions	E300(W)	Chloride Fluoride Sulfate Nitrate Ortho-Phosphate	0.2 0.2 0.2 0.1 0.1	mg/L mg/L mg/L mg/L mg/L	- - - - -
Nitrogen, Nitrate+nitrite	E353.2 (W)	Nitrate+nitrite	0.05	mg/L	-
Petroleum Hydrocarbons	E418.1(W) SW3550 /E418.1(S) SW8015 (Modified)	 Gasoline Diesel, jet fuel	1 1.0 1.0	mg/L mg/L mg/L	30 10 10
1,2-Dibromoethane (EDB)	SW8011		0.05	µg/L	(b)
Arsenic (d)	SW7060 (W&S)		0.005	mg/L	0.5
Lead (d)	SW3005 /SW7421 (W&S)		0.005	mg/L	0.5
Mercury	SW7470 (W) SW7471 (S)		0.001	mg/L	0.1

SYSTEM SRM

STERILE RECORDKEEPING MANAGER

M-SYSTEMS

INTRODUCTION

M-SYSTEMS, INC. specializes in computer application software specifically designed to address the needs of today's hospitals. **M-SYSTEMS'** software has been developed from the ground up in hospitals for ***power, flexibility and affordability***.

Our company's philosophy is based on commitment and dedication to superior products and service. Our product pricing is significantly lower than our competitors' for several reasons. In today's business environment, price is a major issue. By combining industry expertise and maintaining focus, our operational costs are lower, thus the system price to the customer is lower, too. As a result, the customer does not have to sacrifice product quality and service at the expense of price.

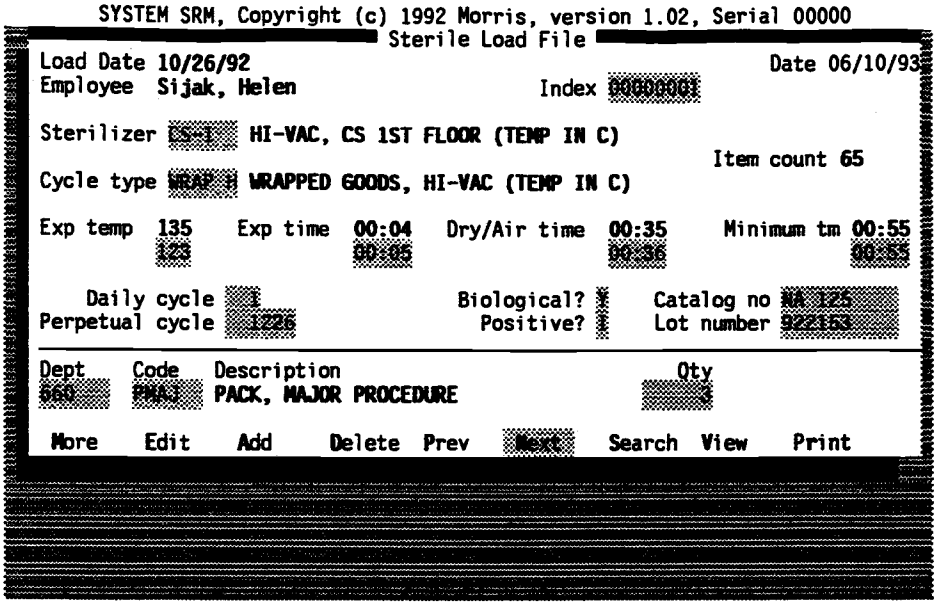
M-SYSTEMS is proud to introduce **SYSTEM SRM - STERILE RECORDKEEPING MANAGER**. This is a totally ***affordable, high performance, user friendly*** Sterile Recordkeeping Manager loaded with features. It is designed to deliver all the features only available, up until now, from system vendors whose prices are, to say the least.....uncomfortable!

SYSTEM SRM offers unsurpassed craftsmanship, a trademark of all our software. It is this quality that sets us apart from our competitors.

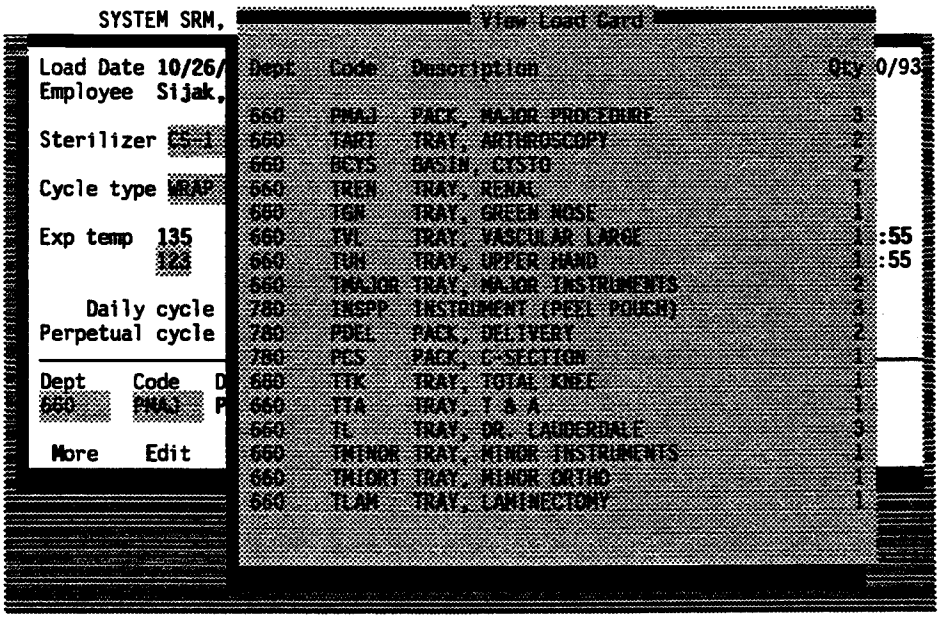
TABLE OF CONTENTS

SYSTEM SRM OVERVIEW

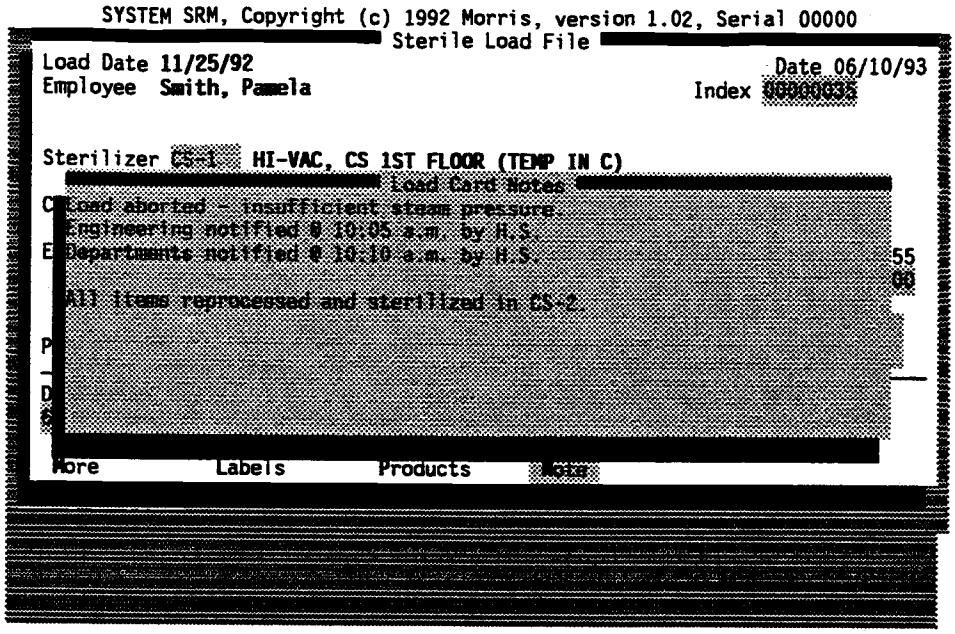
- Eliminate hand written sterilization records by creating them in the system
- Improve the accuracy of your sterilization load records
- Reduce the time it takes to record sterilization information
- Maintain a total number of items in each load at the time the load record is created
- Include a note with any load record (ex: load aborted - not enough steam pressure)
- Print a sterilization record for each load, and/or print a daily loads list of all loads at the end of a day
- Reprint a sterilization record at any time (including the note)
- Locate a load record in the system and view it at any time
- Manage quality assurance information with a touch of a button
- Maintain and print a list of components for every tray, pack and set that you process
- Merge the sterilization information from all Sterile Processing departments in the hospital into ONE quality assurance report
- Print a list of invalid loads (those loads which have incomplete parameters) with a touch of a button for each sterilizer within a specified time frame
- Print a load tally report at a touch of a button for each sterilizer within a specified time frame (this report lists for each sterilizer: a total number of daily cycles, total number of items, total number of tests, total number of biological indicators, total number of positive biological indicators, and total number of invalid loads)
- For ethylene oxide sterilizers, the load tally report shows the ETO exhausted, in pounds, within a specified time frame
- Operate the system on a single computer, or on a network
- When operating on a network, the information is shared among all sterile processing departments in the hospital.



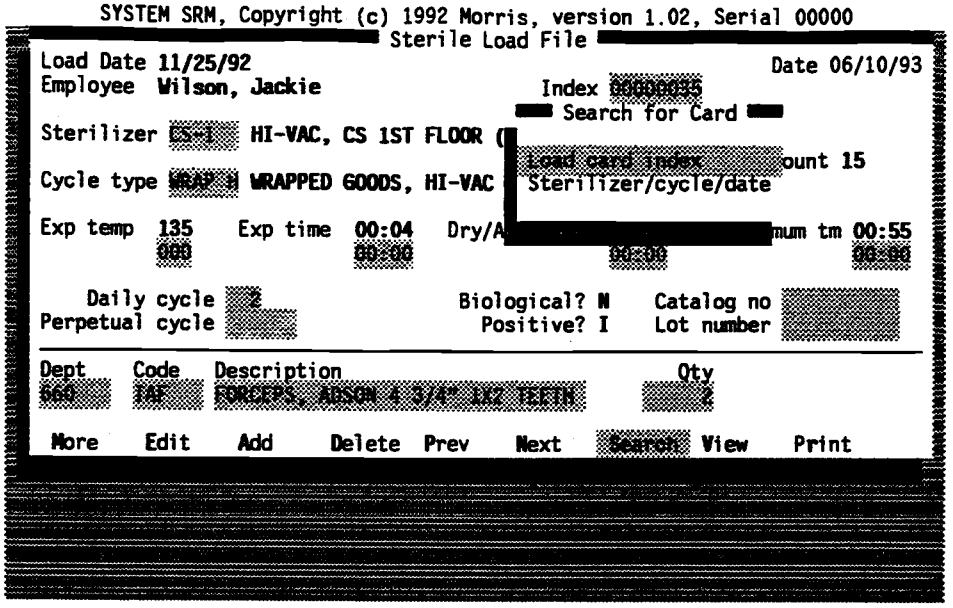
This is a representation of the Sterile Load File screen.



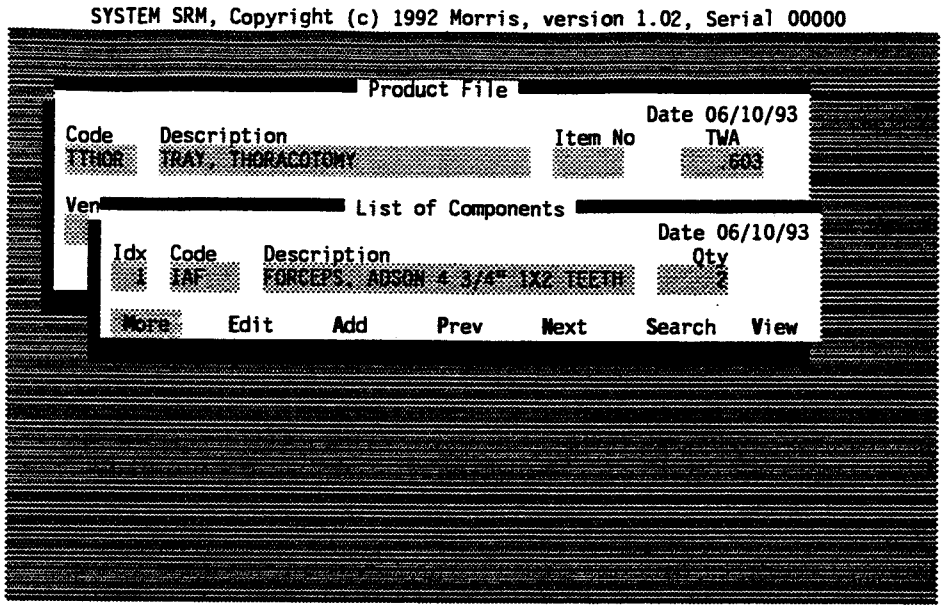
This is a representation of the view screen in the Sterile Load File. You can view the contents of any load card at any time.



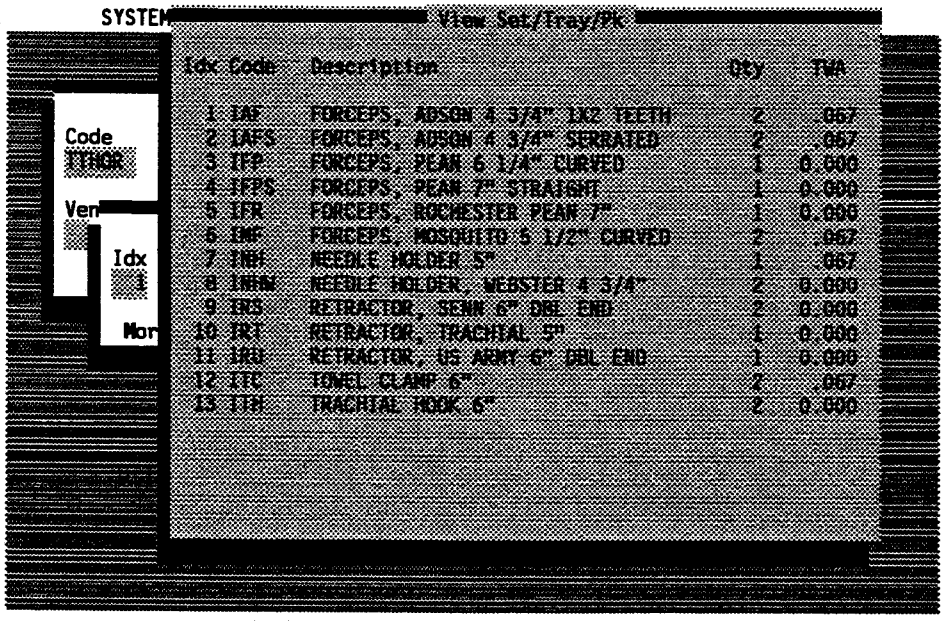
This is a representation of the Load Card Notes screen in the Sterile Load file. You can type notes just like with any other word processor. Help is available by pressing the F1 function key.



You can locate any load record in the system with the search function within seconds.



This is a representation of the Product File screen. This is where you would input all trays, sets, packs and instruments that you process and sterilize for all departments in the hospital. You can also list components for each tray/pack/set that you process. This will be your count sheet to check each tray/pack/set for its completeness.



SYSTEM SRM SAMPLE REPORTS

Date: 02/16/93 Employee: BRDWN,ELEN Index: 0000D037
Sterilizer: CS-1 - HI-VAC, CS 1ST FLDOR (TEMP IN C)
Cycle type: WRAP H- WRAPPED GDDDS, HI-VAC (TEMP IN C)
Exp temp: 135 Exp time: 00:04 Dry/Air: 00:35 Minimum tm: 00:55
Daily cycle: 1 Biological: Y Lot no: 85124 Items: 43

E X P I R A T I D N L A B E L S

Qty	Description	Dept	Qty	Description	Dept
2	FORCEPS, ADSDN 4 3/4" 1X2	432	2	FORCEPS, KELLY 5 1/2" CUR	432
1	TRAY, ALIF	660	2	TRAY, BASIC INSTRUMENTS	660
1	TRAY, T & A	660	1	TRAY, VAGINAL HYSTERECTOM	660
1	TRAY, UPPER HAND	660	2	TRAY, D & C	660
1	PAN, CYSTD	660	1	TRAY, BLUE NDSE	660
2	FORCEPS, ADSDN 4 3/4" 1X2	780	1	TRAY, CUTDOWN ADULT	780
1	TRAY, DEBRIDEMENT	678	1	TRAY, TEETH INSTRUMENTS	678
12	INSTRUMENT (PEEL POUCH)	678	1	FORCEPS, KELLY 5 1/2" CUR	678
2	FDRCEPS, MDSQUITO 5 1/2"	678	2	NEEDLE HOLDER 5"	678
1	TRAY, ARTERIOGRAM	721	2	TRAY, ARTHROGRAM	721
1	TRAY, VASCULAR SMALL	660	1	TRAY, BACK RETRACTORS	660
1	TRAY, TOTAL HIP	660	1	TRAY, DR. LAUDERDALE	660

-01-

The load record contains the necessary cycle and load data. It can be printed at any time. The number of copies is specified by the user. One copy can be enclosed with the load, and another one placed by the sterilizer, so that everyone knows what is being sterilized. The expiration labels can also be included on the record.

Date: 02/16/93 Employee: BROWN,HELEN Index: 0DDD0037
Sterilizer: CS-1 - HI-VAC, CS 1ST FLOOR (TEMP IN C)
Cycle type: WRAP H- WRAPPED GOODS, HI-VAC (TEMP IN C)
Exp temp: 135 Exp time: 00:04 Dry/Air: 00:35 Minimum tm: 00:55
Daily cycle: 1 Biological: Y Lot no: 85124 Items: 43

S T E R I L E L D A D N D T E

Load aborted - insufficient steam pressure.
Engineering notified @ 10:05 a.m. by H.S.
Departments notified @ 10:10 a.m. by H.S.

All items reprocessed and sterilized in CS-2.

A free form load note can be included with any record. Once it becomes a permanent part of the record, it can be printed as needed.

General Hospital
2222 Any Street
Any Town, USA 99999

Page: 001
Printed: 03/25/93

D E P A R T M E N T L I S T

CODE	DEPARTMENT NAME
640	ICU
652	LABOR & DELIVERY
660	OR
662	DAY SURGERY
675	CENTRAL SUPPLY
678	EMERGENCY ROOM
721	RADIOLOGY
780	RHC
TEST	STERILE SUPPLY TEST

The Department list gives you at a glance look of all departments you are sterilizing for.

General Hospital
2222 Any Street
Any Town, USA 99999

Page:001
Printed:03/25/93

S T E R I L I Z E R L I S T

CODE	STERILIZER NAME	SERIAL	MODEL	MFG
CS-1	HI-VAC, CS 1ST FLOOR (TEMP IN C)	Q34212-123	3230	CASTLE
CS-2	HI-VAC, CS 1ST FLOOR - BACKUP UNIT	12254	3230	CASTLE
CS-3	GRAVITY, CS 1ST FLOOR (TEMP IN C)	92263	3220	CASTLE
CS-4	ETHYLENE OXIDE, CS 1ST FLOOR, 100 GR CARTRIDGES (DEG. C)	6554321	400C	3M
LD-1	GRAVITY, L & D 2ND FLOOR (TEMP IN F)	988769	3220	CASTLE
OR-1	FLASH, OR 2ND FLOOR (TEMP IN F) - MAIN UNIT	4403-21220	3210	CASTLE
OR-2	GRAVITY, OR 2ND FLOOR (TEMP IN F) - UNIT IN THE BACK	5544001	2800	AMSCO
OR-3	GRAVITY, OR 2ND FLOOR - BACKUP UNIT	874415	3800	AMSCO

A list of sterilizers located in your department. If System SRM is running on a network, this list will also include sterilizers located in other departments.

complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (e.g., extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured. The analyst will reduce or calculate all raw data into the final reportable values or enter all necessary raw data into the LIMS in order to calculate the final reportable values. Copies of all raw data and the calculations used to generate the final results, such as hardbound laboratory notebooks, strip charts, chromatograms, spreadsheets, and LIMS record files, will be retained to allow reconstruction of the data reduction process at a later date.

For data reporting, rounding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two or three significant figures. In general, formulas for calculations are provided in the field and analytical methods.

8.5.2 Data Review/Validation

The data review process will focus on the following items at a minimum:

- Chain-of-custody forms.
- Holding times.
- Method calibration limits.
- Method blanks.
- Laboratory-established detection and quantitation limits.
- Analytical batch control records, including spike recoveries and duplicate results.
- Corrective actions.
- Formulas used for analyte quantitation.
- Calculations supporting analyte quantitation.
- Completeness of data.

System reviews will be performed at all levels. The individual analyst will constantly review the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews will be performed prior to submission to the Laboratory Manager.

8.5.3 Data Reporting

Reports will contain final results (uncorrected for blanks and recoveries), methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two or three significant figures.

Reported detection limits will account for all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements.

9.0 INTERNAL QUALITY CONTROL CHECKS

9.1 Method Performance QC Indicators: Preparation Batch

Most samples to be analyzed in the laboratory require some pre-treatment before a measurement can be made. Pre-treatment may include extraction, digestion, or distillation. During this step, samples are arranged into discrete manageable groups, called preparation (prep) batches, to facilitate and control uniform treatment for all samples. Each prep batch will have a maximum of 20 investigative samples of the same matrix (e.g., soil or water). In addition, QC indicators such as blanks, spikes, and duplicates are added to each prep batch to monitor the performance of the system. All QC associated with a preparation batch will be carried through the entire analytical procedure, from prep to final analysis.

The preparation blank (PB), also referenced as a method blank (MB) or reagent blank, is used to monitor potential contamination from the sample preparation process. Preparation (prep) blanks will be prepared by processing a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples (when available), through the entire analytical scheme. The reagent blank volume or weight must be approximately equal to the sample volumes or sample weights being processed. In the absence of a suitable solid matrix for soil blanks, reagents will be added to an empty flask and carried through the entire analytical scheme. Results will be calculated based on starting with a "blank" soil approximately equal to the weight of the samples.

9.2 Matrix QC Indicators

Laboratory matrix QC samples are obtained by splitting a field sample into three separate aliquots and performing three separate analyses on the aliquots. Two of the three aliquots will be fortified with target analytes and be designated as a matrix spike and matrix spike duplicate (MS/MSD). The analysis of laboratory spiked duplicates monitors sample precision and accuracy; however, it may be affected by sample inhomogeneity, particularly in the case of nonaqueous samples, as well as reproducibility of laboratory preparation and measurement techniques.

At a minimum one MS/MSD pair will be analyzed for every 20 project samples. Assignment and tracking of the appropriate number of MS/MSD pairs will be done by the QA Manager or client. Samples designated for MS/MSD will be so indicated on the sample bottles, as well as on the chain-of-custody. Since three analyses are required for each MS/MSD, appropriate sample volume must be collected and submitted for analysis.

9.3 Instrument Performance QC Indicators

Instrument performance is monitored each day of use to ensure and document operating conditions conducive to proper target compound identification. The instrument QC indicators appropriate to each analytical technique are identified in the respective method. Refer to the

respective method for applicability.

9.4 Method Performance QC Indicators: Analysis Batch

Matrix-specific QC indicators can be used at the instrument to verify how dependable the measurement is for the respective sample matrix. These indicators provide information on sample matrix effects, which is independent of the efficiency of the preparatory technique. The method performance QC indicators appropriate to each analytical technique are identified in the respective method.

QC checks are performed to provide a tool for evaluating how well the method worked for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project DQOs. For results outside laboratory control limits, appropriate corrective action will be taken and the deviation noted in the case narrative accompanying the sample results.

9.5 QC Monitoring - Laboratory QC Charts

The analyses of quality control samples are entered chronologically onto quality control charts specifically maintained for each analytical procedure. These control charts are labeled with upper and lower warning limits corresponding to $\pm$ two times the standard deviation of the mean of the accumulated results, and upper and lower control limits corresponding to $\pm$ three times the standard deviation of the mean of the accumulated results, analysis which is being charted, and the value (e.g., % recovery, % RPD, etc.) which is being monitored.

QC charts and associated control limits are updated quarterly and are used to demonstrate method performance and help identify system errors. More frequent QC charts can be generated monthly or even daily based on the most recent 40 data points in order to track any potential out-of-control QC trends for a given parameter.

Laboratory internal quality control procedures, frequency, acceptance criteria and corrective actions are summarized in Table 9-1.

9.6 Performance and Systems Audits

Quality assurance audits and surveillances are conducted to verify conformance with the laboratory's quality assurance program, to determine the effectiveness of the QA program, and to continually improve the quality effort.

Performance audits test the laboratory's ability to correctly assay an unknown sample. They may be single blind or double blind. In a single blind study, the analyst is not provided with the acceptable results for the unknown sample until after the experiemental result is reported; however, it is known that the sample is a performance test. In a double blind performance test, the analyst not only has no knowledge of the acceptable result, but the sample is

Table 9-1
Summary of Internal Quality Control Procedures

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ E	Metals/ Inorganics	Method Blank	1 per analytical batch of samples, not to exceed 20 samples of a given matrix	<3x reporting limit for all analytes	• Reanalyze to determine if instrument contamination was the cause.
					• If a sample contains target compounds at > 10X amount found in the method blank, or if target compounds are <u>not</u> detected in the samples, then that sample does not require redigestion and the results may be reported without qualifications.
					• If the method blank is still noncompliant and the samples are within the digestion holding time, then redigest and reanalyze all associated samples containing target compounds at, 10X amount found in the method blank and reporting limit.
					• If the samples are outside the digestion holding time, then contact the project manager using the Sample Discrepancy Report (SDR).
		Laboratory Control Sample (LCS)	1 per analytical batch of ≤ 20 samples of a given matrix	See Table 3-1	• Check calculations and spike preparation for documentable errors.
					• If no errors are found, then reanalyze the LCS to determine if instrumental conditions or analytical preparation was the cause.
					• If the LCS are still noncompliant, and the samples are within the digestion holding time, then re-digest and reanalyze all associated samples.
					• If the samples are outside the digestion holding time, initiate an SDR. Contact the project manager for sample disposition.
		Initial Calibration Verification Standard (ICV)	Once immediately following the calibration curve	90% - 100% for metals except mercury; 80 - 120% for mercury	If mis-rack or mis-prep of ICVs are suspected, remake, rerack, and reanalyze on same curve starting with ICV; otherwise, recalibrate and rerun.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ E	Metals/ Inorganics	Initial Calibration Verification Bank (ICB)	Once immediately following the ICV	< 3X reporting limit (Table 8-1)	If ICB is high, but samples nondetect, narrate and report. If mis-rack or mis-prep of ICBs are suspected, remake, rerack, and reanalyze on same curve starting with ICV; otherwise, recalibrate and rerun.
		Continuing Calibration Verification Standard (CCV)	1 per 10 samples analyzed	90 - 100% for all metals except mercury; 80 - 120% for mercury	Rerun the samples for the last good CCV/CCB. Recalibrate or reslope if necessary.
		Continuing Calibration Verification Blank (CCB)	1 per 10 samples analyzed	< 3X reporting limit (Table 8-1)	Rerun the samples after the last good CCV/CCB. Recalibrate or reslope if necessary. If CCB is high but samples nondetect, narrate and report.
		Linear Range Check Standard (low level): Metals only	After initial calibration (CRII) and (for ICP only) at the end of the analytical sequence (CRIF) or every 8 hours	Not established	
		Interference Check Sample (ICSAB): ICP only	After calibration and at the end of the analytical sequence	80 - 120% recovery	Rerun and/or recalibrate
		Serial Dilution (L): ICP only	1 per 20 project samples per matrix	<10% difference if original sample result is >50X IDL	Advisory only. Note in narrative. Evaluate for usability
		Matrix Spike (MS)	1 per 20 project samples per matrix	See Table 3-3	Advisory only. Note in Narrative. Evaluate for usability.
		Matrix Spike Duplicate (MSD)	1 per 20 project samples per matrix	See Table 3-3	Advisory only. Note in narrative. Evaluate for usability.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ E	Metals/ Inorganics	Method of Standard Addition (MSA); GFAA only	If the single analytical spike is >40% and sample absorbance is >50% of spike absorbance and the 85%> recovery <115%	Correlation coefficient: ≥0.995	Rerun once. Report data with best correlation.
		Coefficient of Variation: (Metals only)	All multiple injections	±20% RPD	If the concentration is >RL; rerun once.
SW846/8260	Organics VOA	Tuning Criteria	Every 12-hour period	BFB mass spectrum	Retune. Do not proceed with analysis until tune meets criteria.
		Initial Calibration	When CCCs and SPCCs in the daily calibration do not meet criteria	CCC: ±30% RSD; SPCC: ≥0.30, except bromoform ≥0.25	Reanalyze the initial calibration curve and/or evaluate/correct instrument malfunction to obtain curve which meets criteria.
		Daily Calibration	Every 12-hour period following tune	CCC: ±25 D SPCC: ≥0.30, except bromoform ≥0.25	Reanalyze the daily standard. If still out, notify supervisor. Evaluate/correct instrument malfunction as needed; initiate a new calibration curve.
		Method Blank	Every 12-hour period after each calibration	≤3X the reporting limit for all analytes (Table 8-1)	Reanalyze to determine if instrument contamination was the cause. If the method blank is still noncompliant, correct the problem before analysis of samples.
		Matrix Spikes (MS) Matrix Spike Duplicate (MSD)	1 per 20 project samples per matrix	See Table 3-3	The recoveries for the spiked compounds should be within advisory limits. If noncompliant, check calculations and spike preparation for documentable errors. If no errors are found, analyze blank spike. If the associated blank spikes are within advisory limits, then sample matrix effects are the most likely cause. Note in narrative. Evaluate for usability.
		Blank spikes (BS)	1 for each MS/MSD outside control limits	See Table 3-3	Notify the Supervisor and initiate corrective action. Reanalyze associate samples, if appropriate.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ 8260	Organics VOA	Surrogate Spikes	Every sample	See Table 3-2	Reanalyze sample once. If still out, report results and note in narrative. Evaluate for usability.
		Internal Standard (IS)	Every sample, standard, and blank	Average area within -50% to + 100% window	Inspect instrument for malfunction; correct identified malfunctions, then reanalyze samples. if no malfunction, evaluate for usability: • If surrogate recoveries also out of criteria, reanalyze once and report (IS areas affect recoveries). • If surrogates meet criteria and IS areas are >5000 area counts or >25% of the VSTPS IS areas, then analysis is not required. • If out-of-limit areas are explained by the sample matrix, reanalysis will not be required (e.g, high hydrocarbon content contributes to IS areas.) • For multiple analyses (MS, MSD, rep.), sample dilutions fulfill reanalysis requirements.
			Every sample, standard, and blank	RT shift <30 seconds compared to daily standard (STD50)	Inspect chromatographic system for malfunction; correct identified malfunctions,then reanalyze sample.
SW 846/ 8270	Organics BNA	Tuning Criteria	Every 12-hour period	DFTPP mass spectrum	Retune. Do not proceed with analysis until tune meets criteria.
		Initial Calibration	When CCCs and SPCCs in the daily calibration do not meet criteria	CCC: +30% RSD SPCC: >0.05	Reanalyze the initial calibration curve and/or evaluate/correct instrument malfunction to obtain curve which meets criteria.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ 8270	Organics BNA	Daily Calibration	Every 12-hour period following tune	CCC: +25% D SPCC: >0.05	Reanalyze the daily standard. If still out, notify Supervisor. Evaluate/correct instrument malfunction as needed; initiate a new calibration curve.
		Method Blank	1 per each preparation batch of ≤20 samples of the same matrix	≤3X the reporting limit for all analytes (Table 8-1)	Reanalyze to determine if instrument contamination was the cause. If the method blank is still noncompliant, initiate correction action (SDR): • If a sample contains target compounds at ≥10X amount found in the method blank, or if target compounds are not detected in the sample, then that sample does not require reextraction and the results may be reported without qualifications. • If the samples are within the extraction holding time, then reextract and reanalyze all associated samples containing target compounds at <10X amount found in the method blank. • If the samples are outside the extraction holding time, then contact the project manager using the Sample Discrepancy report (SDR) for sample disposition.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ 8270	Organics BNA	Matrix Spike (MS) Matrix Spike Duplicate (MSD)	1 per 20 project samples per matrix	See Table 3-3	• If recoveries for the spiked compounds are not within advisory limits, check for documentable errors (e.g, calculations and spike preparation). If no errors are found, and the associated blank spikes are within advisory limits, then sample matrix effects are the most likely cause. Note in narrative. Evaluate for usability. • If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects.
		Blank Spikes (BS)	1 for each MS/MSD outside control limits	See Table 3-3	Notify the Supervisor and initiate corrective action (SDR). Evaluate impact on data. Reextract/reanalyze associated samples. • If holding times are an issue, notify the PM for sample disposition.
		Surrogate Spikes	Every Sample	See Table 3-2 1 surrogate from each fraction may be outside criteria; however, none may be < 10% recovery	Initial Corrective Action (SDR) • If the surrogate recoveries in the associated method blank and blank spike are <u>not</u> within limits, and the samples are within holding time, re-extract and reanalyze the affected samples once. If still out, report results and note in narrative. Evaluate for usability. • If holding times are an issue, notify the PM for sample disposition.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ 8270	Organics BNA	Internal Standard (IS)	Every sample, standard, and blank	Average area within -50% to + 100% window	Inspect instrument for malfunction; correct identified malfunctions, then reanalyze samples. If no satisfaction, evaluate for usability: • If surrogate recoveries also out of criteria, reanalyze once and report (IS areas affect recoveries). • If surrogates meet criteria and IS areas are >5,000 area counts or >25% of the STD50 IS areas, then reanalysis is not required. • If out-of-limit areas are explained by the sample matrix, reanalysis will not be required (e.g, high hydrocarbon content contributes to IS areas). • For multiple analyses (MS, MSD, rep), sample dilutions, and reextracted analyses fulfill re-analysis requirements.
		Retention Time Shift (RT)	Every sample, standard, and blank	RT shift <30 seconds compared to daily standard (STD50)	Inspect chromatographic system for malfunction; correct identified malfunctions, then reanalyze sample.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ GC Methods	Organics Pest PCBs, Herbicides, Volatile Aromatic, etc.	Initial Calibration	As needed, i.e., when the daily calibration does not meet criteria	±20% RSD for the calibration factors (CF) over the range	Reanalyze the initial calibration curve and/or evaluate correct instrument malfunction to obtain curve that meets criteria.
		Daily Calibration (ICV) and Continuing Calibration (CCV)	At the beginning of the daily sequence (when full curve not analyzed); and 1 CCV every 10 samples	±15% D in CF from the corresponding initial curve standard on at least one column for each target compound; and on both columns for any confirmed compound	• If more than 10% of the target compounds fail this criteria, then the daily calibration standard is considered to be noncompliant. Reanalyze ICV. • If ICV still noncompliant, evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a new standard). • If ICV still noncompliant, recalibrate the instrument with a new curve. • Samples analyzed after a failed CCV will be reanalyzed. • If a failed CCV (e.g., for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported; and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. • If holding times are an issue, complete an SDR and notify the PM for sample disposition.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ GC Methods		Retention Time Shift (RT)	10%, using the RT of Analyses in the CCV	Column and compound-specific; varies with each ICV	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate. Evaluate date based on a comparison with other standards run during the analytical sequence, consider the RT's for the surrogates and spiked compounds analyzed before and after the sample in question: • Expand the RT windows to encompass the shift in compound location • If no peaks are found in the expanded window, report the compound as nondetect. • If peaks are present, use the confirmation column to verify identification.

Table 9-1
Summary of Internal Quality Control Procedures (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW 846/ GC Methods		Method Blank	1 per each preparation batch of $\leq$20 samples of the same matrix	$\leq$3X the reporting limit for all analytes (Table 8-1)	Reanalyze to determine if instrument contamination was the cause. If the method blank is still non-complaint, initiate corrective action: • If a sample contains target compounds at > 10X amount found in the method blank, or if target compounds are not detected in the sample, then that sample does not require reextraction and the results may be reported without qualifications. • If the samples are within the extraction holding time, then reextract and reanalyze all associated samples containing target compounds at < 10X amount found in the method blank. • If the samples are outside the extraction holding time, then contact the project manager using the Sample Discrepancy Report (SDR) for sample disposition.
		Matrix Spike (MS) Matrix Spike (MSD)	1 MS/MSD per 20 project samples per matrix	See Table 3-3	• If recoveries for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and the associated blank spikes are within advisory limits, then sample matrix effects are the most likely cause. Note in narrative. Evaluate for usability.

disguised in such a manner as to maintain anonymity as a performance test sample.

Systems audits and surveillances evaluate the operational details of the QA program. An audit consists of a systematic procedure to ascertain the implementation of a specific QA requirement, such as sample tracking or chain-of-custody procedures. Audits will be conducted by persons other than those who performed or directly supervised the work being inspected. A surveillance consists of inspection or monitoring of a specific targeted area for compliance to requirements, such as an evaluation of a single analytical method to ensure conformance with the written protocol.

9.6.1 External Audits

Performance audits, as well as on-site systems audits, by external agencies and clients are an ongoing occurrence. A full response to any deficiencies cited as a result of a performance audit or on-site visit will be addressed within a timely manner, as determined by the client. The QA Manager is responsible for scheduling and coordinating all external audits.

Performance Audits

Single blind performance audits (i.e., Performance Evaluation Studies (PES)) are routinely analyzed at least four times a year: twice a U.S. Environmental Protection Agency (EPA) WS study and twice an EPA WP study. Additionally, air matrix NIOSH PAT samples are analyzed quarterly.

PES samples are additionally analyzed as double and single blinds on a project-specific basis for selected clients, including the Corps of Engineers, federal and state agencies, as well as nongovernment clients.

Systems Audits

On-site evaluation by agencies, clients, or designated their party auditors (both government and nongovernment) are routinely conducted to ensure program compliance.

9.6.2 Internal Audits

The laboratory QA Manager has overall responsibility for monitoring the internal QA/QC program.

Performance Audits

Internal performance audits conducted at the bench level provide the analyst with a tool to self-evaluate the acceptability of a specific data set. This is accomplished through analysis of laboratory control samples or spiked blanks of known concentration to the analyst, which must meet minimum performance standards.

10.0 DATA ASSESSMENT PROCEDURES

The definitions of precision, accuracy, and completeness are discussed in Subsection 1.4. All analytical data will be reviewed relative to these criteria and specific project requirements to assess the quality of the analytical data. Where all criteria are met, data will be deemed acceptable without qualification. Where precision and accuracy goals are not met, the sample set will be reanalyzed or reported with qualification in the case narrative. Some of the factors affecting this final sample disposition will include:

- Project-specific QA/QC requirements.
- Availability of sufficient sample for reanalysis.
- Holding time considerations.
- Regulatory action limits.

10.1 Precision

Precision is measured through analysis of replicate QC controls and field samples. Results from these measurements are calculated as relative percent difference (percent RSD) or percent relative standard deviation (percent SD) and evaluated according to the criteria set forth in Subsection 3.2. Laboratory QC control samples are used to demonstrate acceptable method performance and are used to trigger corrective action when control limits are exceeded. Precision requirements for organic analyses are listed in Tables 3-1 and 3-3.

Precision measurements from field samples give an indication of sample homogeneity. Problems with sample homogeneity are more likely to occur with soil samples. Percent differences for field samples are advisory only. Corrective action is triggered only by RPD for standard matrix samples (e.g., blanks).

10.2 Accuracy

Accuracy is measured through the analysis of fortified LCS and fortified field samples. Results from these measurements are calculated as percent recovery and evaluated for accuracy as described in Subsection 3.1. QA/QC criteria are presented in Tables 3-1, 3-2, and 3-3. Laboratory QC control samples (spiked blanks, LCSs) are used to demonstrate acceptable method performance and are used to trigger corrective action when control limits are exceeded. Accuracy requirements for selected organic surrogate recoveries are listed in Table 3-2.

Surrogates are generally isotopically labeled analytes (or other compounds not normally found in the environment) that are chemically similar to target analytes. They are added in known amounts prior to analysis to check the method performance on individual samples.

Accuracy measurements from field samples give an indication of physical or chemical interferences present that can either enhance or mask the actual presence of target analytes.

Determination of percent recovery (percent R) requires analysis of a fortified sample and a nonfortified sample, so that any background analyte already present in the sample can be accounted for in the recovery determination. Thus, sample homogeneity also becomes a factor in recovery determinations, as variable background can affect the apparent analyte recovery. Problems with sample recovery are more likely to occur with soil and sediment samples, water samples containing a noticeable amount of solids, and nonstandard matrices. Recovery data for field samples are only advisory. Only data for standard matrix samples (blanks) are used to trigger corrective action.

10.3 Completeness

Completeness is defined as a measure of the amount of analytical data meeting all accuracy and precision criteria generated by an analytical method or system. The minimum goal for completeness is 90 percent, and the ability to exceed this goal is dependent on the applicability of the analytical methods to the sample matrices analyzed. However, even if data have not met this laboratory definition of data able to be reported without qualification, project completion goals may still be met if the qualified data, i.e., data of known quality even if not perfect, are suitable for specified project goals.

10.4 Laboratory Internal Data Review/Validation

The QA Manager reviews data to ensure consistency with laboratory QC requirements, to verify reasonableness with other generated data, and to determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made concerning whether the analysis should be repeated. In addition, the QA Manager will recalculate selected results to verify the calculation procedure.

Prior to final review/sign off by the Laboratory Manager, the QA Manager will verify that the report deliverable is complete and in proper format, screen the report for compliance to laboratory and client QA/QC requirements, and ensure that the case narrative covers any noted deficiencies. The Laboratory Manager will be the final laboratory review prior to reporting the results to the client's Project Manager.

Data audits are also performed by regulatory agencies, client representatives, or third party data validators. The frequency, level of detail and the areas of concern during these reviews depend on specific program requirements.

As an additional feature of the laboratory's internal QA Program, double blind performance evaluation samples are periodically submitted to the laboratory for analysis. These samples originate both internally and externally, and are scheduled through the laboratory's project management system to ensure anonymity. Over the course of a year, samples are submitted to cover all routinely analyzed methods.

Systems Audits and Surveillances

Internal laboratory systems audits and surveillances are conducted and documented on a quarterly basis, at a minimum. Each quarter's audit will target a limited section of the laboratory and be coordinated such that the entire laboratory is planned for QA audit at least once annually. The internal audit consists of a review of laboratory systems, procedures, and documentation. Any deficiencies and/or deviations are documented and a summary report prepared.

9.7 Preventive Maintenance

The ability to generate valid analytical data requires that all analytical instrumentation be properly and regularly maintained. The responsibility of routine care lies with the analysts using the instruments. Guidance on required routine maintenance, as well as troubleshooting information, is provided in the respective instrument manuals and laboratory SOPs. For more extensive preventative maintenance or emergency repair service, the analytical laboratory maintains full-service contracts on all major instruments. The elements of the maintenance program are discussed in the following subsections.

9.7.1 Instrument Maintenance Logbooks

Each analytical instrument is assigned an instrument logbook. All maintenance activities are recorded in the instrument log. The information entered in the instrument log includes:

- Date of service or maintenance
- Person performing service or maintenance
- Type of service performed and reason for service
- Replacement parts installed (if appropriate)
- Documentation of the reestablishment of working order
- Miscellaneous information.

9.7.2 Instrument Maintenance and Repair

Preventative maintenance and repairs that cannot be performed by laboratory staff are contracted to the manufacturer's service department or to an authorized maintenance vendor. Chemron service agreements provide for preventative maintenance, emergency service, and emergency shipping of spare parts. Annual service of the laboratory balances is an example of contracted preventative maintenance.

SYSTEM SRM

STERILE RECORDKEEPING MANAGER

M-SYSTEMS

INTRODUCTION

M-SYSTEMS, INC. specializes in computer application software specifically designed to address the needs of today's hospitals. **M-SYSTEMS'** software has been developed from the ground up in hospitals for ***power, flexibility and affordability***.

Our company's philosophy is based on commitment and dedication to superior products and service. Our product pricing is significantly lower than our competitors' for several reasons. In today's business environment, price is a major issue. By combining industry expertise and maintaining focus, our operational costs are lower, thus the system price to the customer is lower, too. As a result, the customer does not have to sacrifice product quality and service at the expense of price.

M-SYSTEMS is proud to introduce **SYSTEM SRM - STERILE RECORDKEEPING MANAGER**. This is a totally ***affordable, high performance, user friendly*** Sterile Recordkeeping Manager loaded with features. It is designed to deliver all the features only available, up until now, from system vendors whose prices are, to say the least.....uncomfortable!

SYSTEM SRM offers unsurpassed craftsmanship, a trademark of all our software. It is this quality that sets us apart from our competitors.

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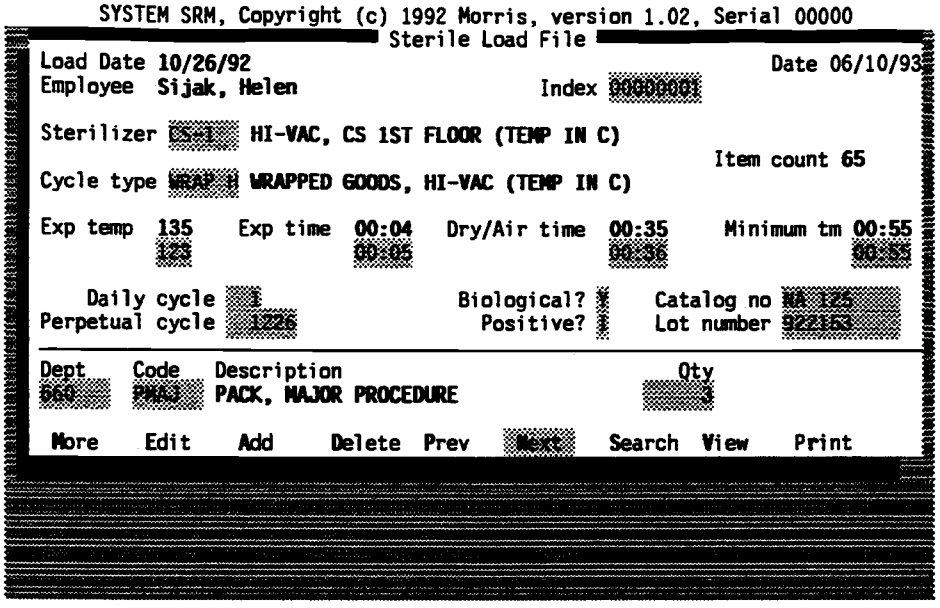
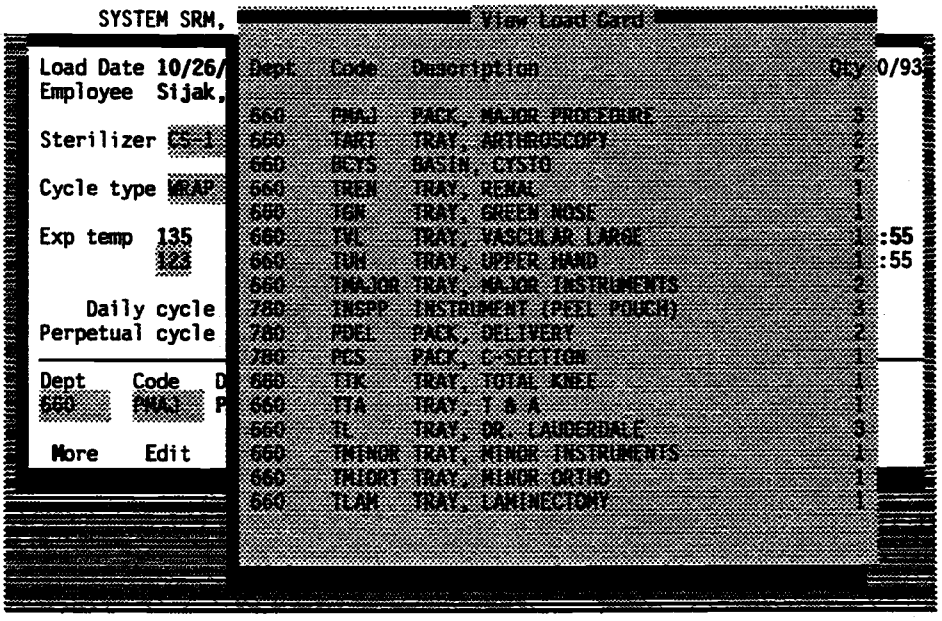
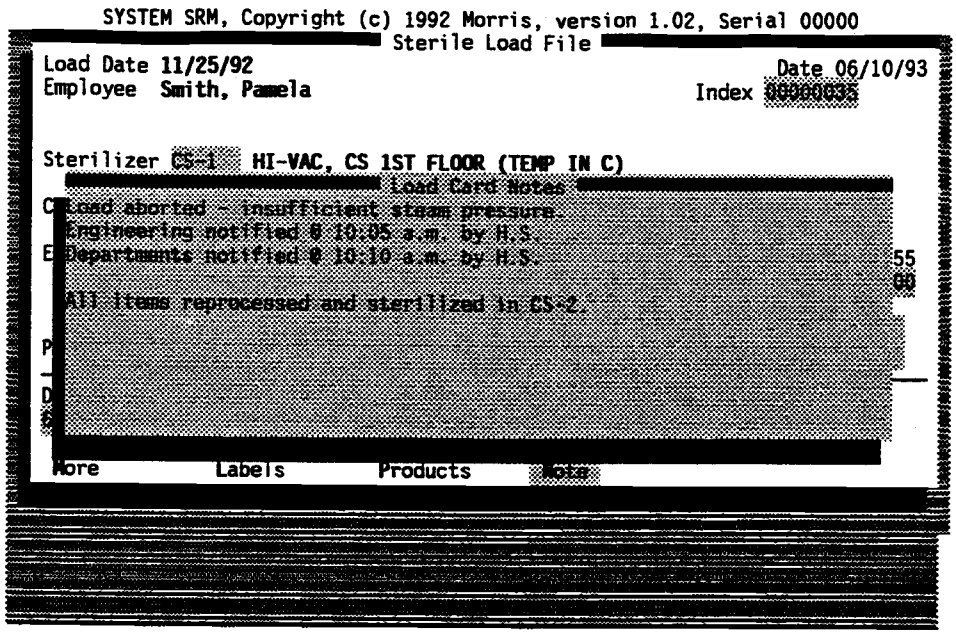
- Eliminate hand written sterilization records by creating them in the system
- Improve the accuracy of your sterilization load records
- Reduce the time it takes to record sterilization information
- Maintain a total number of items in each load at the time the load record is created
- Include a note with any load record (ex: load aborted - not enough steam pressure)
- Print a sterilization record for each load, and/or print a daily loads list of all loads at the end of a day
- Reprint a sterilization record at any time (including the note)
- Locate a load record in the system and view it at any time
- Manage quality assurance information with a touch of a button
- Maintain and print a list of components for every tray, pack and set that you process
- Merge the sterilization information from all Sterile Processing departments in the hospital into ONE quality assurance report
- Print a list of invalid loads (those loads which have incomplete parameters) with a touch of a button for each sterilizer within a specified time frame
- Print a load tally report at a touch of a button for each sterilizer within a specified time frame (this report lists for each sterilizer: a total number of daily cycles, total number of items, total number of tests, total number of biological indicators, total number of positive biological indicators, and total number of invalid loads)
- For ethylene oxide sterilizers, the load tally report shows the ETO exhausted, in pounds, within a specified time frame
- Operate the system on a single computer, or on a network
- When operating on a network, the information is shared among all sterile processing departments in the hospital.



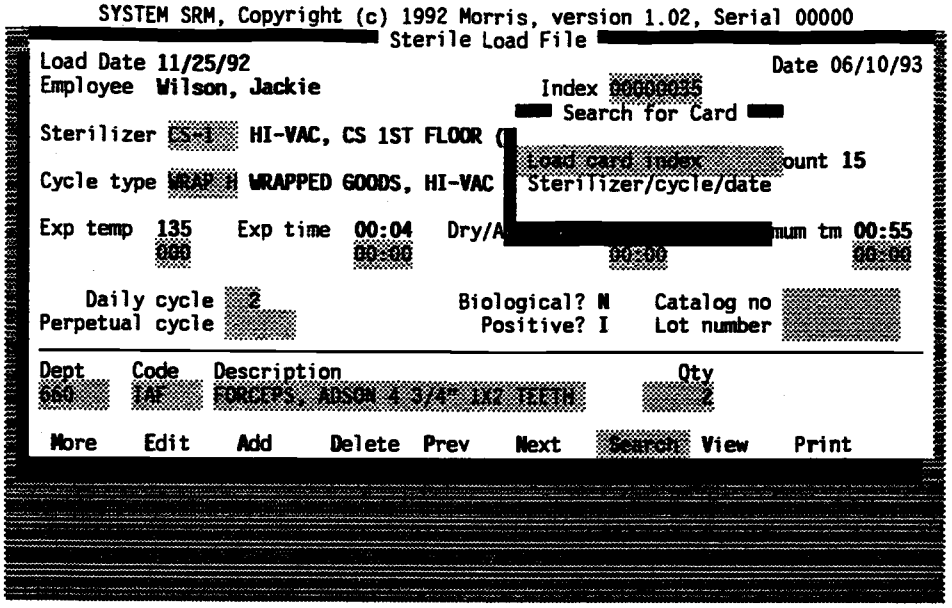
This is a representation of the Sterile Load File screen.



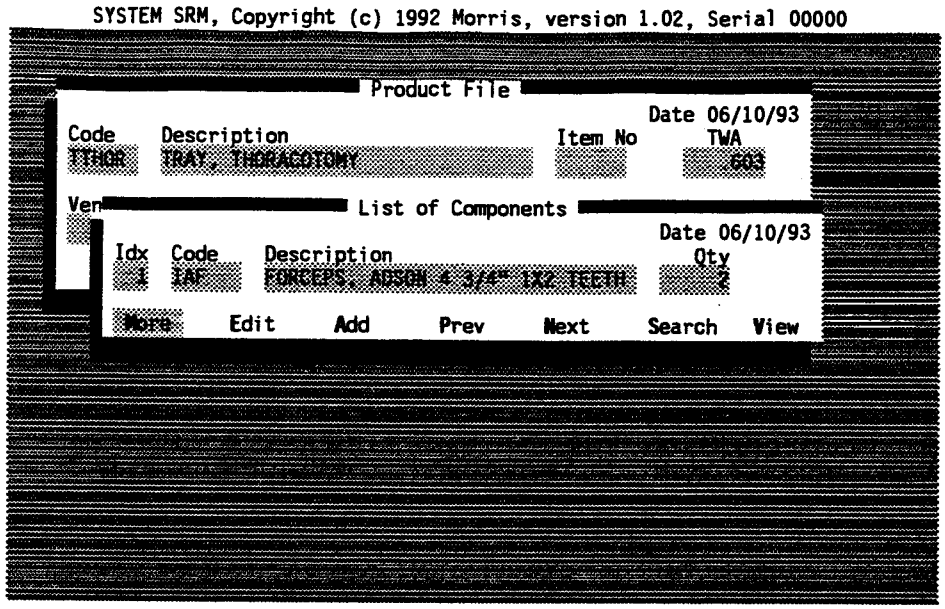
This is a representation of the view screen in the Sterile Load File. You can view the contents of any load card at any time.



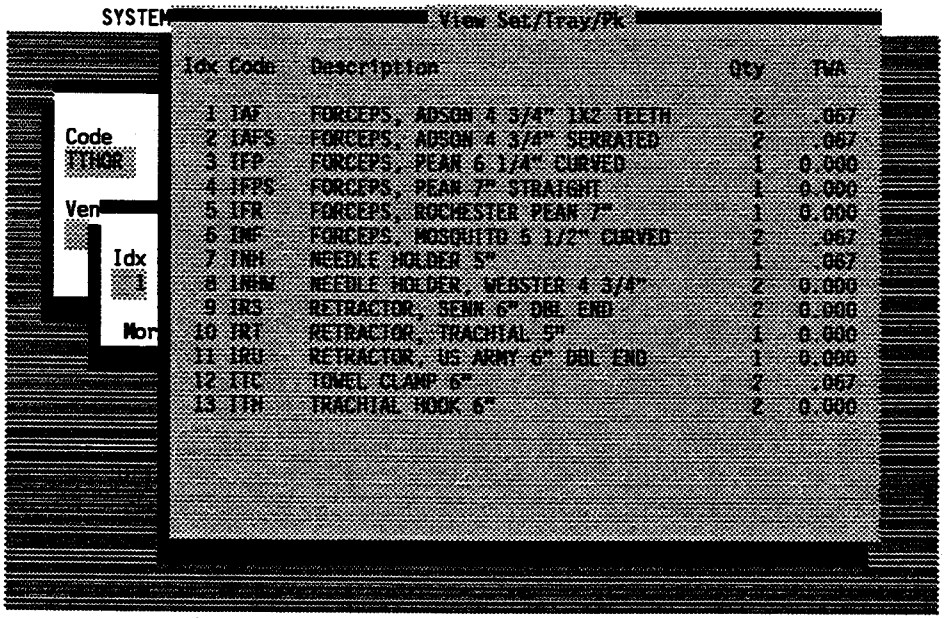
This is a representation of the Load Card Notes screen in the Sterile Load file. You can type notes just like with any other word processor. Help is available by pressing the F1 function key.



You can locate any load record in the system with the search function within seconds.



This is a representation of the Product File screen. This is where you would input all trays, sets, packs and instruments that you process and sterilize for all departments in the hospital. You can also list components for each tray/pack/set that you process. This will be your count sheet to check each tray/pack/set for its completeness.



SYSTEM SRM SAMPLE REPORTS

Date: 02/16/93 Employee: BROWN,ELEN Index: 00000037
Sterilizer: CS-1 - HI-VAC, CS 1ST FLOOR (TEMP IN C)
Cycle type: WRAP H- WRAPPED GOODS, HI-VAC (TEMP IN C)
Exp temp: 135 Exp time: 00:04 Dry/Air: 00:35 Minimum tm: 00:55
Daily cycle: 1 Biological: Y Lot no: 85124 Items: 43

E X P I R A T I O N L A B E L S

Qty	Description	Dept	Qty	Description	Dept
2	FORCEPS, ADSON 4 3/4" 1X2	432	2	FORCEPS, KELLY 5 1/2" CUR	432
1	TRAY, ALIF	660	2	TRAY, BASIC INSTRUMENTS	660
1	TRAY, T & A	660	1	TRAY, VAGINAL HYSTERECTOM	660
1	TRAY, UPPER HAND	660	2	TRAY, D & C	660
1	PAN, CYSTO	660	1	TRAY, BLUE NOSE	660
2	FORCEPS, ADSON 4 3/4" 1X2	780	1	TRAY, CUTDOWN ADULT	780
1	TRAY, DEBRIDEMENT	678	1	TRAY, TEETH INSTRUMENTS	678
12	INSTRUMENT (PEEL POUCH)	678	1	FORCEPS, KELLY 5 1/2" CUR	678
2	FORCEPS, MOSQUITO 5 1/2"	678	2	NEEDLE HOLDER 5"	678
1	TRAY, ARTERIOGRAM	721	2	TRAY, ARTHROGRAM	721
1	TRAY, VASCULAR SMALL	660	1	TRAY, BACK RETRACTORS	660
1	TRAY, TOTAL HIP	660	1	TRAY, DR. LAUDERDALE	660

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The load record contains the necessary cycle and load data. It can be printed at any time. The number of copies is specified by the user. One copy can be enclosed with the load, and another one placed by the sterilizer, so that everyone knows what is being sterilized. The expiration labels can also be included on the record.

Date: 02/16/93 Employee: BROWN,HELEN Index: 00000037
Sterilizer: CS-1 - HI-VAC, CS 1ST FLOOR (TEMP IN C)
Cycle type: WRAP H- WRAPPED GOODS, HI-VAC (TEMP IN C)
Exp temp: 135 Exp time: 00:04 Dry/Air: 00:35 Minimum tm: 00:55
Daily cycle: 1 Biological: Y Lot no: 85124 Items: 43

S T E R I L E L O A D N O T E

Load aborted - insufficient steam pressure.
Engineering notified @ 10:05 a.m. by H.S.
Departments notified @ 10:10 a.m. by H.S.

All items reprocessed and sterilized in CS-2.

A free form load note can be included with any record. Once it becomes a permanent part of the record, it can be printed as needed.

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D E P A R T M E N T L I S T

CODE	DEPARTMENT NAME
640	ICU
652	LABOR & DELIVERY
660	OR
662	DAY SURGERY
675	CENTRAL SUPPLY
678	EMERGENCY ROOM
721	RADIOLOGY
780	RHC
TEST	STERILE SUPPLY TEST

The Department list gives you at a glance look of all departments you are sterilizing for.

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S T E R I L I Z E R L I S T

CODE	STERILIZER NAME	SERIAL	MODEL	MFG
CS-1	HI-VAC, CS 1ST FLOOR (TEMP IN C)	Q34212-123	3230	CASTLE
CS-2	HI-VAC, CS 1ST FLOOR - BACKUP UNIT	12254	3230	CASTLE
CS-3	GRAVITY, CS 1ST FLOOR (TEMP IN C)	92263	3220	CASTLE
CS-4	ETHYLENE OXIDE, CS 1ST FLOOR, 100 GR CARTRIDGES (DEG. C)	6554321	400C	3M
LD-1	GRAVITY, L & D 2ND FLOOR (TEMP IN F)	988769	3220	CASTLE
OR-1	FLASH, OR 2ND FLOOR (TEMP IN F) - MAIN UNIT	4403-21220	3210	CASTLE
OR-2	GRAVITY, OR 2ND FLOOR (TEMP IN F) - UNIT IN THE BACK	5544001	2800	AMSCO
OR-3	GRAVITY, OR 2ND FLOOR - BACKUP UNIT	874415	3800	AMSCO

A list of sterilizers located in your department. If System SRM is running on a network, this list will also include sterilizers located in other departments.

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CYCLE PARAMETER LIST

CODE	CYCLE PARAMETER	TEMP	EXP TIME	DRY/AIR	MINTIME
B&D	BOWIE & DICK TEST CYCLE (TEMP IN C)	135	00:03	00:01	00:12
B&DF	BOWIE & DICK CYCLE (TEMP IN F)	270	00:03	00:00	00:20
COLD	COLD	37	05:15	08:30	13:45
ETO W	WARM (12/88 MIXTURE)	55	06:00	08:00	14:00
FLASH	FLASH	250	00:07	00:00	00:10
GRAV	WRAPPED GOODS , GRAVITY (TEMP IN F)	250	00:30	00:40	01:18
LIQ	LIQUID	250	00:20	00:00	00:40
WARM	WARM	55	02:22	08:30	10:52
WRAP	WRAPPED GOODS, HI-VAC (TEMP IN F)	270	00:06	00:50	00:56
WRAP G	WRAPPED GOODS, GRAVITY (TEMP IN C)	121	00:30	00:40	01:18
WRAP H	WRAPPED GOODS, HI-VAC (TEMP IN C)	135	00:04	00:35	00:55

A list of each different cycle you run in each of your sterilizers.

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PRODUCT LIST

CODE	PRODUCT NAME	TWA
BCYS	BASIN, CYSTO	
DRILL	DRILL, POWER	0.125
DRILLD	DRILL, DENTAL	0.698
IAF	FORCEPS, ADSON 4 3/4" 1X2 TEETH	0.067
IAFS	FORCEPS, ADSON 4 3/4" SERRATED	0.067
IFKC	FORCEPS, KELLY 5 1/2" CURVED	
IFKS	FORCEPS, KELLY 5 1/2" STRAIGHT	
IFP	FORCEPS, PEAN 6 1/4" CURVED	
IMF	FORCEPS, MOSQUITO 5 1/2" CURVED	0.067
IMFS	FORCEPS, MOSQUITO 5 1/2" STRAIGH	0.067
INH	NEEDLE HOLDER 5"	0.067
INHW	NEEDLE HOLDER, WEBSTER 4 3/4"	
INSPP	INSTRUMENT (PEEL POUCH)	0.067
LENSD	LENS, LAPAROSCOPE DOUBLE PUNCT	
LENSS	LENS, LAPAROSCOPE SINGLE PUNCT	
PCYS	PAN, CYSTO	
REAM	REAMER, POWER	
SAW	SAW, MICRO SAGITAL POWER	
TALIF	TRAY, ALIF	0.134
TBRET	TRAY, BACK RETRACTORS	
TCA	TRAY, CUTDOWN ADULT	1.273
TCIR	TRAY, CIRCUMCISION	
TCRAN	TRAY, CRANIOTOMY	
TCS	TRAY, C-SECTION	
TDC	TRAY, D & C	
TFR	TRAY, FRAGMENT	
TH	TRAY, TOTAL HIP	
THY	TRAY, HYSTERECTOMY	
THYS	TRAY, HYSTEROSCOPY	
TLAM	TRAY, LAMINECTOMY	
TLDPI	TRAY, LAP DOUBLE PUNCTURE INSTR.	
TLHER	TRAY, LAP HERNIA	
TLSPI	TRAY, LAP SINGLE PUNCTURE INSTR.	
TMAJOR	TRAY, MAJOR INSTRUMENTS	
TMINOR	TRAY, MINOR INSTRUMENTS	
TMIORT	TRAY, MINOR ORTHO	
TMJORT	TRAY, MAJOR ORTHO	
TMYR	TRAY, MYRINGOTOMY	
TUH	TRAY, UPPER HAND	
TVS	TRAY, VASCULAR SMALL	

To get a listing of all trays, packs, sets, instruments and miscellaneous items you process, print a Product List. It will also include the TWA (time weighted average) for each item.

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D A I L Y L O A D S

10/27/92 to 10/27/92

Sterilizer: CS-1 Daily cycle: 1 Date: 10/27/92 TWA: 0.000

6 TRAY, MAJOR INSTRUMENTS 660

Sterilizer: CS-2 Daily cycle: 1 Date: 10/27/92 TWA: 1.742

11 INSTRUMENT (PEEL POUCH) 675
12 INSTRUMENT (PEEL POUCH) 678
3 INSTRUMENT (PEEL POUCH) 640

Sterilizer: CS-3 Daily cycle: 1 Date: 10/27/92 TWA: 1.216

3 BASIN, CYSTO 660
1 TRAY, MINOR ORTHO 678
2 TRAY, TOTAL HIP 678
2 DRILL, POWER 678
2 TRAY, ARTHROSCOPY 660
4 INSTRUMENT (PEEL POUCH) 678
2 TRAY, MAJOR INSTRUMENTS 678
3 TRAY, T & A 678
1 DRILL, DENTAL 660

Sterilizer: CS-3 Daily cycle: 3 Date: 10/27/92 TWA: 2.429

2 TRAY, ARTERIOGRAM 721
5 INSTRUMENT (PEEL POUCH) 721
3 DRILL, DENTAL 662

Sterilizer: CS-4 Daily cycle: 1 Date: 10/27/92 TWA: .469

2 LENS, LAPAROSCOPE DOUBLE 660
3 INSTRUMENT (PEEL POUCH) 660
2 INSTRUMENT (PEEL POUCH) 743
2 LENS, LAPAROSCOPE SINGLE 660
2 INSTRUMENT (PEEL POUCH) 678

Sterilizer: CS-4 Daily cycle: 2 Date: 10/27/92 TWA: .402

6 INSTRUMENT (PEEL POUCH) 660
2 LENS, LAPAROSCOPE SINGLE 660
2 LENS, LAPAROSCOPE DOUBLE 660

Sterilizer: LD-1 Daily cycle: 1 Date: 10/27/92 TWA: 4.891

3 PACK, C-SECTION 652
1 PACK, OB 652
1 TRAY, CIRCUMCISION 652
2 PACK, DELIVERY 652
4 INSTRUMENT (PEEL POUCH) 652
1 TRAY, DELIVERY 652

Instead of printing individual load records, you can print Daily Loads list at the end of each day. You can attach the autoclave charts to this list and file it.

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INVALID LOADS

10/01/92 to 10/31/92

Sterilizer	Cycle	Date	Reason(s)
CS-1	1	10/26/92	Temp
CS-1	3	10/26/92	Temp, Exp tm, Dry/air tm, Total tm
CS-2	1	10/26/92	Temp, Exp tm, Dry/air tm, Total tm
CS-2	2	10/26/92	Temp, Exp tm, Dry/air tm, Total tm
CS-3	2	10/26/92	Temp, Exp tm, Dry/air tm, Total tm
CS-3	3	10/26/92	Temp, Exp tm, Dry/air tm, Total tm
CS-1	1	10/27/92	Temp, Exp tm, Total tm
CS-2	1	10/27/92	Temp, Exp tm, Dry/air tm, Total tm
CS-3	1	10/27/92	Temp, Exp tm, Dry/air tm, Total tm
CS-3	3	10/27/92	Temp, Exp tm, Dry/air tm, Total tm
CS-4	1	10/27/92	Exp tm, Dry/air tm, Total tm
CS-4	2	10/27/92	Temp, Exp tm, Dry/air tm, Total tm
LD-1	1	10/27/92	Temp, Exp tm, Dry/air tm, Total tm
CS-1	1	10/29/92	Temp, Exp tm, Dry/air tm, Total tm
CS-3	1	10/29/92	Temp, Exp tm, Dry/air tm, Total tm
CS-4	1	10/29/92	Exp tm, Dry/air tm, Total tm

To locate loads that have not met sterilization parameters, print the Invalid Loads list. Use this list to troubleshoot your sterilizers.

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LOAD TALLY REPORT

10/01/92 to 12/31/92

Sterilizer	Cycles	Items	Tests	Bio	Positive	Invalid	Chg LB
CS-1	13	260	3	4	0	9	0.000
CS-2	3	50	0	0	0	3	0.000
CS-3	9	168	0	2	0	8	0.000
CS-4	5	53	0	5	0	3	1.102
ER-1	1	3	0	0	0	1	0.000
LD-1	2	18	0	1	0	1	0.000

The Load Tally Report provides the user with the pertinent quality assurance information. The time frame for which the information is needed is user definable. This information is always available within seconds.

For ETO sterilizers, this report lists the total ETO charge (in pounds) within a specified time frame.

TRAY, THORACOTOMY COUNT SHEET (TTHOR)

PAR	COUNT	DESCRIPTION	PAR	COUNT	DESCRIPTION
2	_____	FORCEPS, ADSON 4 3/4" 1X2	2	_____	FORCEPS, ADSON 4 3/4" SER
1	_____	FORCEPS, PEAN 6 1/4" CURV	1	_____	FORCEPS, PEAN 7" STRAIGHT
1	_____	FORCEPS, ROCHESTER PEAN 7	2	_____	FORCEPS, MOSQUITO 5 1/2"
1	_____	NEEDLE HOLDER 5"	2	_____	NEEDLE HOLDER, WEBSTER 4
2	_____	RETRACTOR, SENN 6" DBL EN	1	_____	RETRACTOR, TRACHIAL 5"
1	_____	RETRACTOR, US ARMY 6" DBL			

Total Instruments:_____

CS Tech:_____ Date:_____

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Once you list the components for each tray/set/pack, you can print the count sheets. The standard count sheet would be used by Sterile Supply personnel when they are assembling the trays.

TRAY, THORACOTOMY COUNT SHEET (TTHOR)

DESCRIPTION	PAR	CS COUNT	OR COUNT	NUMBER ADDED	CS COUNT
FORCEPS, ADSON 4 3/4" 1X2	2	_____	_____	_____	_____
FORCEPS, ADSON 4 3/4" SER	2	_____	_____	_____	_____
FORCEPS, PEAN 6 1/4" CURV	1	_____	_____	_____	_____
FORCEPS, PEAN 7" STRAIGHT	1	_____	_____	_____	_____
FORCEPS, ROCHESTER PEAN 7	1	_____	_____	_____	_____
FORCEPS, MOSQUITO 5 1/2"	2	_____	_____	_____	_____
NEEDLE HOLDER 5"	1	_____	_____	_____	_____
NEEDLE HOLDER, WEBSTER 4	2	_____	_____	_____	_____
RETRACTOR, SENN 6" DBL EN	2	_____	_____	_____	_____
RETRACTOR, TRACHIAL 5"	1	_____	_____	_____	_____
RETRACTOR, US ARMY 6" DBL	1	_____	_____	_____	_____

CS Tech:_____ Date:_____

OR:_____ Date:_____

CS Tech:_____ Date:_____

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The extended count sheet would be used by Sterile Supply personnel and Surgery personnel to check the completeness of the tray/set/pack, and include any added instruments.